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Guidelines for the quality control of measurements of particulate emissions from stationary sources by the Federal reference method are presented. These include:

1. Good operating practices,
2. Directions on how to assess performance and qualify data,
3. Directions on how to identify trouble and improve data quality,
4. Directions to permit design of auditing activities.

The document is not a research report. It is designed for use by operating personnel.

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GUIDELINES FOR DEVELOPMENT OF A QUALITY ASSURANCE PROGRAM: VOLUME IV - DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES

by

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ABSTRACT

Guidelines for the quality control of measurements of particulate emissions from stationary sources by the Federal reference method are presented. These include:

1. Good operating practices,
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3. Directions on how to identify trouble and improve data quality,
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The document is not a research report. It is designed for use by operating personnel.

This work was submitted in partial fulfillment of Contract Durham 68-02-1234 by Research Triangle Institute under the sponsorship of the Environmental Protection Agency. Work was completed as of February 1975.

SECTION I

INTRODUCTION

SECTION I INTRODUCTION

This document presents guidelines for developing a quality assurance program for Method 5, Determination of Particulate Emissions from Stationary Sources. This method was published by the Environmental Protection Agency in the Federal Register, December 23, 1971. The method was revised in January 1975 and the revised method* is reproduced as Appendix A of this report for convenience of reference.

~~This document~~ is divided into four sections:

Section I, Introduction. The introduction lists the overall objectives of a quality assurance program and delineates the program components necessary to accomplish the given objectives.

Section II, Operations Manual. This manual sets forth recommended operating procedures to insure the collection of data of high quality and instructions for performing quality control checks designed to give an indication or warning that invalid data or data of poor quality are being collected, allowing for corrective action to be taken before future measurements are made.

Section III, Manual for Field Team Supervisor. This manual contains directions for assessing data quality on an intralaboratory basis and for collecting the information necessary to detect and/or identify trouble.

Section IV, Manual for Manager of Groups of Field Teams. This manual presents information relative to the test method (a functional analysis to identify the important operations variables and factors, and statistical properties of and procedures for carrying out auditing procedures for an independent assessment of data quality).

The objectives of this quality assurance program for Method 5 are to:

1. Identify and where possible eliminate systematic errors (biases) and maintain precision within acceptable limits in the measurement process,
2. Provide routine indications for operating purposes of satisfactory performance of personnel and/or equipment,
3. Provide for prompt detection and correction of conditions that contribute to the collection of poor quality data,

cont

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* The revised method became available just prior to the printing of this document. Consequently, there are numerous duplications of the material as given in Appendix A and in the Operations Manual of this document.

4. Collect and supply information necessary to describe the quality of the data.

To accomplish the above objectives, a quality assurance program must contain the following components:

1. Recommended operating procedures,
2. Routine training of personnel and evaluation of performance of personnel and equipment,
3. Routine monitoring of the variables and parameters that may have a significant effect on data quality,
4. Development of statements and evidence to qualify data and detect defects,
5. Action strategies to increase the level of precision/accuracy in the reported data.

Component 2 above will be treated for all the methods in the final report of this contract. All other components are treated in this document.

Implementation of a properly designed quality assurance program should enable measurement teams to achieve and maintain an acceptable level of precision and accuracy in their stack gas-composition measurements. It will also allow a team to report an estimate of the precision of its measurements for each source emissions test.

Variability in emission data derived from multiple tests conducted at different times includes components of variation from:

1. Process conditions,
2. Equipment and personnel variation in field procedures,
3. Equipment and personnel variation in the laboratory.

In many instances time variations in source output may be the most significant factor in the total variability. This component of variation is minimized by knowing the time characteristics of the source output and maintaining isokinetic sampling conditions. The sampling period should span at least one complete output cycle, if an average over the cycle is desired. If the cycle is too long and an average value is desired, either the sample collection should be made during a portion of the cycle representative of the cycle average, or multiple samples should be collected and averaged.

Quality assurance guidelines for Method 5 as presented here are designed to insure the collection of data of acceptable quality by prevention, detection, and quantification of equipment and personnel variations in both the field and the laboratory through:

1. Recommended operating procedures as a preventive measure,
2. Quality control checks for rapid detection of undesirable performance,
3. A quality audit to independently verify the quality of the data.

The scope of this document has been purposely limited to that of a field and laboratory document. Additional background information is contained in the final report under this contract.

SECTION II OPERATIONS MANUAL

2.0 GENERAL

This manual sets forth recommended procedures for determination of particulate emissions from stationary sources according to Method 5. (Method 5 as revised by EPA is included as Appendix A of this document.) Quality-control procedures and checks are designed to give an indication or warning as part of the operating procedures and are to be performed by the operator on a routine basis. In addition, the performance of special quality control procedures and/or checks as prescribed by the supervisor for assurance of data quality may be required of the operator on special occasions.

The sequence of operations to be performed for each field test is given in Figure 1. Each operation or step in the method is identified by a block. Quality checkpoints in the measurement process, for which appropriate quality-control limits are assigned, are represented by blocks enclosed by heavy lines. Other quality checkpoints involve go/no-go checks and/or subjective judgments by the test team members with proper guidelines for decisionmaking spelled out in the procedures.

The precision/accuracy of data obtained from this method depends upon equipment performance and on the proficiency and conscientiousness with which the operator performs his various tasks. From equipment checks through on-site measurements, calculations, and data reporting, this method is susceptible to a variety of errors. Detailed instructions are given for minimizing or controlling equipment error, and procedures are recommended in order to minimize operator error. Before using this document, the operator should study Method 5 as reproduced in Appendix A in detail. In addition, the quality assurance documents of this series for Methods 2, 3, and 4 (refs. 1, 2 and 3) should be read and followed. Also, these methods, i.e., methods 2, 3 and 4, have been extensively revised by the Environmental Protection Agency in an effort to improve the quality of the data obtained by field teams using these methods. The methods as

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EQUIPMENT SELECTION

1. SELECT EQUIPMENT ACCORDING TO THE GUIDELINES GIVEN IN SUBSECTION 2.1 FOR THE SOURCE TO BE TESTED.

EQUIPMENT CALIBRATION

2. CALIBRATE EQUIPMENT ACCORDING TO SUBSECTION 2.2.

PRESAMPLING PREPARATION

3. OBTAIN PROCESS DATA, SELECT/PREPARE SAMPLING SITE, DETERMINE LOGISTICS FOR PLACING EQUIPMENT ON-SITE, AND DETERMINE STACK CONDITIONS T_s, P_s B_{ws}, AND M_s. (SUBSECTION 2.3.1)

4. CHECK OUT SAMPLING TRAIN AND RELATED COMPONENTS. (SUBSECTION 2.3.2)

5. PACKAGE AND SHIP EQUIPMENT. (SUBSECTION 2.3.3)

ON-SITE MEASUREMENTS

6. MOVEMENT OF EQUIPMENT TO SAMPLING SITE AND SAMPLE RECOVERY AREA. (SUBSECTION 2.4.1)

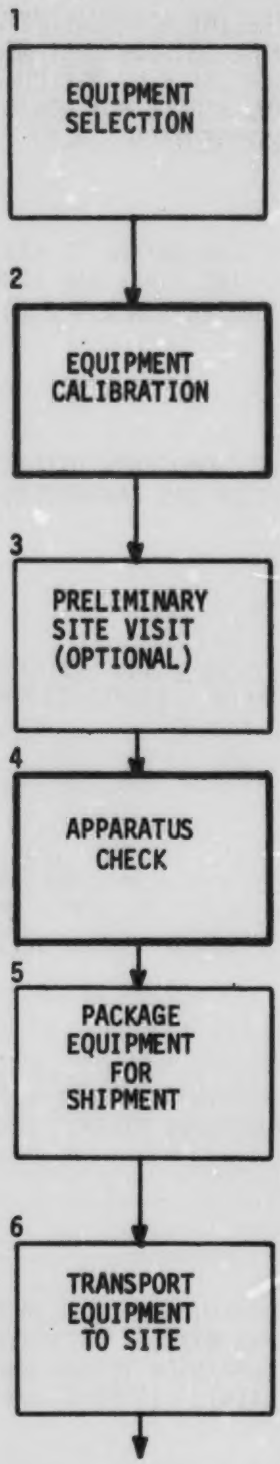

1
EQUIPMENT SELECTION
2
EQUIPMENT CALIBRATION
3
PRELIMINARY SITE VISIT (OPTIONAL)
4
APPARATUS CHECK
5
PACKAGE EQUIPMENT FOR SHIPMENT
6
TRANSPORT EQUIPMENT TO SITE

Figure 1. Operational flow chart of the measurement process.

7. PRELIMINARY MEASUREMENTS AND SETUP WILL INCLUDE DUCT MEASUREMENT, PERCENT H_2O, MOLECULAR WEIGHT (M_s), AND SAMPLE BOX LOGISTICS. (SUBSECTION 2.4.2)

8. DETERMINATION OF MAXIMUM AND MINIMUM ΔP AND STACK GAS TEMPERATURE. (SUBSECTION 2.4.2.5)

9. SET NOMOGRAPH UTILIZING THE PRELIMINARY STACK GAS PARAMETERS [APPENDIX B].

10. ASSEMBLE AND LEAK CHECK THE SAMPLING TRAIN. (SUBSECTIONS 2.4.3.3 THROUGH 2.4.3.6)

11. COLLECT A MINIMUM SAMPLE VOLUME OF 1.7 m^3 60 FT^3. MAINTAIN ISOKINETIC CONDITIONS DURING SAMPLING. (SUBSECTION 2.4.3.7)

12. PERFORM FINAL LEAK CHECK OF THE ENTIRE SAMPLING TRAIN. (SUBSECTION 2.4.3.7(7))

13. OBTAIN SAMPLE FROM FILTER HOLDER, PROBE AND NOZZLE. MEASURE TOTAL CONDENSATE (IMPINGER VOLUME AND SILICA GEL WEIGHT GAIN). (SUBSECTION 2.4.4)

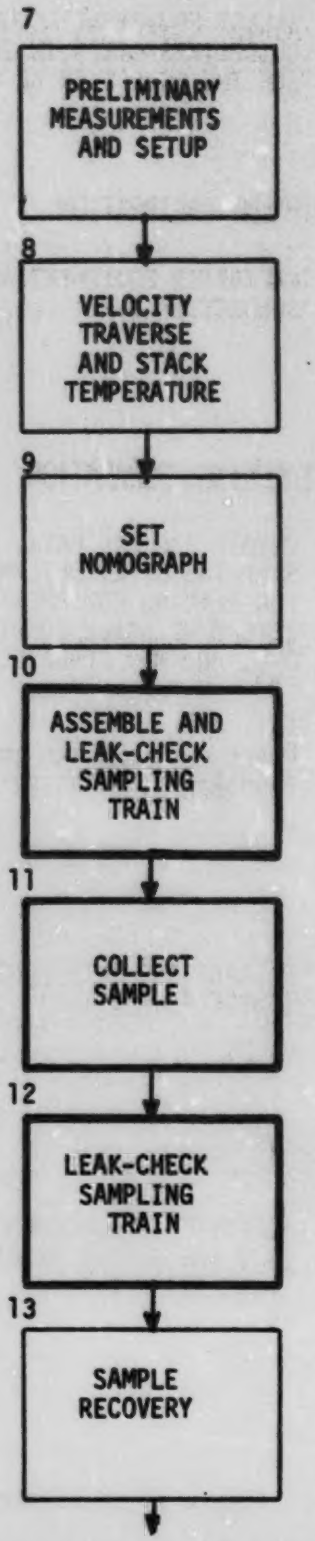

7
PRELIMINARY MEASUREMENTS AND SETUP
8
VELOCITY TRAVERSE AND STACK TEMPERATURE
9
SET NOMOGRAPH
10
ASSEMBLE AND LEAK-CHECK SAMPLING TRAIN
11
COLLECT SAMPLE
12
LEAK-CHECK SAMPLING TRAIN
13
SAMPLE RECOVERY

Figure 1. Operational flow chart of the measurement process (continued).

14. VALIDATE THE DATA BY CHECKING ALL DATA SHEETS FOR COMPLETENESS. COMPARE MEASURED VALUES OF V_s, B_{ws}, AND M_s WITH THEORETICAL VALUES. (SUBSECTION 2.4.6)

15. DISASSEMBLE AND INSPECT EQUIPMENT FOR DAMAGE SUSTAINED BUT NOT DETECTED DURING SAMPLING (SUBSECTION 2.4.6)

16. PACKAGE EQUIPMENT FOR RETURN TRIP TO BASE LABORATORY. (SUBSECTION 2.4.6)

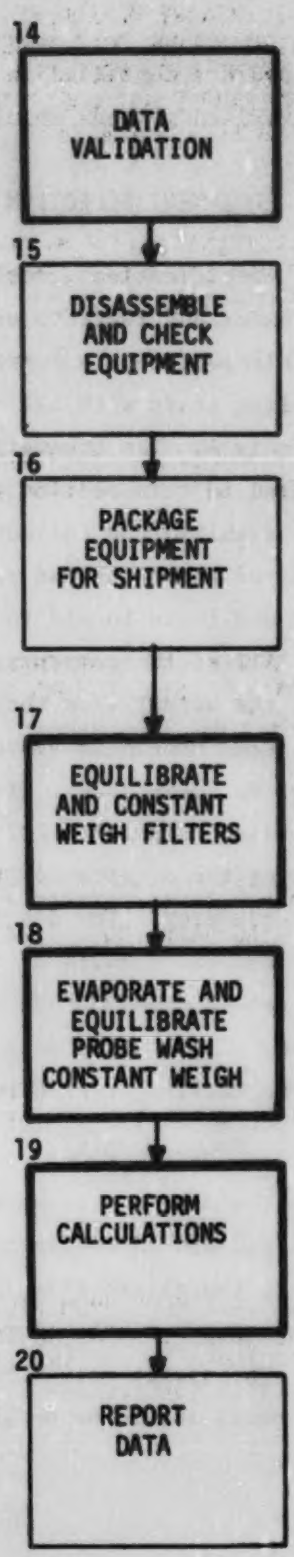
POSTSAMPLING OPERATIONS

17. DESICCATE FILTERS AND CONSTANT WEIGH TO ± 0.5 mg. RECORD WEIGHT GAIN TO THE NEAREST 0.1 mg. (SUBSECTION 2.5.2)

18. TRANSFER ACETONE WASHING TO TARED BEAKER, EVAPORATE AT AMBIENT CONDITIONS, DESICCATE TO A CONSTANT WEIGHT AND RECORD DATA TO THE NEAREST 0.1 mg. (SUBSECTION 2.5.2)

19. PERFORM NECESSARY CALCULATIONS TO OBTAIN PARTICULATE CONCENTRATION, VOLUMETRIC FLOW RATE, AND PERCENT ISOKINETIC VARIATION. (SUBSECTION 2.5.3)

20. FORWARD THE DATA FOR FURTHER INTERNAL REVIEW OR TO THE USER.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DATA VALIDATION
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DISASSEMBLE AND CHECK EQUIPMENT
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PACKAGE EQUIPMENT FOR SHIPMENT
17
EQUILIBRATE AND CONSTANT WEIGH FILTERS
18
EVAPORATE AND EQUILIBRATE PROBE WASH CONSTANT WEIGH
19
PERFORM CALCULATIONS
20
REPORT DATA

Figure 1. Operational flow chart of the measurement process (continued).

revised are contained in the Final Report under this contract (ref 4). The revised methods should be followed in future field tests.

2.1 EQUIPMENT SELECTION

Specifications, design features, and acceptance criteria for the equipment and reagents used in measuring particulate emissions are given in this section. A corresponding schematic of an assembled particulate sampling train with all components identified is shown in Figure 5-1 of Appendix A. The schematic will aid in the comprehension of the material covered in this section which will insure proper selection of equipment and, finally, the collection of high quality data. A listing of the required apparatus and reagents, along with certain miscellaneous equipment and tools to aid in source testing, is given in this section.

All of the components described in this section should be procured with the stipulation that they meet the criteria discussed below. All new items should be visually inspected for identification and damage prior to acceptance. If applicable, new equipment should be calibrated according to section 2.2 as part of the acceptance check. Equipment not meeting the acceptance standards must be rejected or modified to meet the standards. The descriptive title, any applicable identification number, and the results of the acceptance check are recorded in the receiving record file, dated, and signed by the individual performing the check. Calibration data generated in the acceptance check are recorded in the calibration log book.

2.1.1 Probe Nozzle

The nozzle serving as an interface between the sampling probe and the flue gas should result in a minimum of disturbance to the flow pattern and to the particulate concentration. The nozzle must be inert to the sample gases at the temperatures encountered in the field.

The nozzle should be thin-walled with a beveled, sharp leading edge. The bevel should be on the outside with a continuous, smooth inside surface; i.e., a constant internal diameter must be preserved. A button-hook shaped nozzle (see Figure 5-1 of Appendix A) is required to allow for easy insertion through small ports when sampling a thick-walled stack.

A set containing a minimum of three nozzles with 6.4-, 9.5-, and 13- mm (1/4-, 3/8-, and 1/2-in) inside diameters is recommended. Low stack gas velocity, high moisture content, or high stack gas temperature may require 14- or 16- mm (9/16 or 5/8 in) diameter nozzles in order to achieve isokinetic sampling. These sizes are not generally stock items, but are available from the manufacturer upon request.
The following specifications are recommended:

Material:	
Temperatures below 870°C (1600°F)	Seamless stainless steel (316) tubing for 2- to 3-hour exposures (ref. 5).
Temperatures above 870°C (1600°F)	Quartz
Temperatures up to 1370°C (2500°F)	Nickel-base alloys such as Inconel (subject to severe corrosion in the presence of fluorine or chlorine)
Wall Thickness:	1.65 mm (0.065 in.)
Angle of Taper:	≤ 30°
Distance from Tip of Nozzle to First Bend:	At least two times the outside nozzle diameter
Inside Diameter:	Available in increments of 1.6 mm (1/16 in.)
Probe Nozzle Brush:	See section 2.1.2.

2.1.2 Sampling Probe

The sampling probe should be made of stainless steel or of borosilicate (Pyrex) glass and encased in a steel sheath. The probe must be equipped with a heating system that will maintain an even (preset) temperature along the length of the probe to avoid condensation and reevaporation of the sample gas. The probe material must be non-reactive with the gas constituents so as not to introduce a bias into the analytical method. Because of this, a knowledge of the stack gas composition and temperature is necessary to select the correct probe.

A new probe should be visually checked for identification, i.e., verify that it is the length and composition ordered. The probe should be checked for cracks or breaks and leak-checked on a sampling train as described in subsection 2.3.2.1(2). Also, the probe heating system should be calibrated according to subsection 2.2.2. Any probe not satisfying the acceptance check should be repaired, if possible or rejected.

A probe brush of an appropriate size and shape is necessary to clean the probe liner and nozzle prior to and after sampling.

The following specifications are suggested for the probe and probe brush:

Probe Material:	
Temperature $\leq$ 480°C (900°F)	Thick-walled borosilicate glass or quartz glass
Temperature $\leq$ 900°C (1650°F)	Quartz
Inside Diameter:	1.3 cm (.5 in.) approximately
Heating System:	
Temperature requirements at the exit and within 0.3 m (12 in) of the inlet	No greater than 120 ± 14 °C (248 ± 25°F) with a gas flow of 0.02 m^3/min (.75 ft^3/min) at room temperature
Probe Brush:	
Bristles	Nylon
Handles	Stainless steel wire
Extensions	Inert material and total length should be at least as long as the probe.

2.1.3 Filter Holder

The filter holder should be durable, easy to load, and leak-free in normal applications. The design must be such that the filter material is not torn as the holder is tightened. Also, the only flow through the holder must be through the filter. New filter holders are checked visually for cracks and sharp edges that could puncture or tear a filter. The filter holder with a filter installed is placed in a sampling train and leak-checked at about 380 mm Hg (15 in. Hg) vacuum as directed in subsection 2.4.3.5. Disassemble the holder and check the filter for punctures and cuts. Reject the holder if any of the checks are negative and cannot be corrected.

The glass frit should always be cleaned before sampling according to the mnaufacturer's recommendations. A standard cleaning procedure is not applicable to all filter supports due to the variation in construction materials, but this construction material should allow the rinsing of the interior surface of the filter holder and frit with 1:1 (V/V) hydrochloric acid and water. This wash should be followed with a final rinse of distilled, deionized water.

Specifications for the filter holder are:

Material:	Borosilicate (Pyrex) glass (other materials may be used if approved by the EPA administration)
Leak Sealant:	Non-reactive to the stack gases
Filter Support Media:	Glass frit

2.1.4 Filter Holder Box

The filter box must be equipped with a heating system and a temperature monitoring device to regulate the temperature around the filter holder during sampling. A dial-type thermometer is recommended for monitoring the temperature.

Visually check the filter holder box for damage. Check the heating system by calibrating it as directed in subsection 2.2.2. Reject the box if it is damaged or if a preset temperature in the range of 120 ± 14°C cannot be maintained. The heating system and thermometer should meet the following criteria:

Desired Temperature:	No greater than 120 ± 14°C (248 ± 25°F) in ambient temperatures down to 0°C (32°F)

Thermometer:

Accuracy	± 3°C (± 5°F)
Range	16° to 150°C (60° to 300°F)

2.1.5 Impingers/Condenser/Container

The sample gas, having passed through the filter, must be cooled to 20°C (68°F) or less, and the moisture content of the stack gas measured. To accomplish this task, four impingers are connected in series. The first, third and fourth impingers (see Figure 2) are of the modified Greenburg-Smith design. The second impinger is of the Greenburg-Smith design with the standard tip. Each impinger is checked visually for damage, such as breaks or cracks, and manufacturing flaws, such as poorly shaped connections. The container should be checked for damage and filled with water to see if it leaks. Reject any item that is faulty.

The fourth impinger is required (charged with 200 g of preweighed silica gel) to remove moisture and to protect the vacuum pump and dry gas meter. The reference method requires that the condensate trap be used to keep the effluent gas temperature at 20°C (68°F) or less. Ice containers are available in commercial trains or can be fabricated from closed-pore expanded polyethylene. The efficiency of the ice bath can be increased by the addition of salt.

Specifications are as follows:

Second Impinger:	Tip must be large enough to allow an impinger full of water to drain in at least 6 to 8 seconds (this avoids an excessive pressure drop in the sampling system)
Modified Impingers:	Replace tip with a 13 mm (1/2 in.) diameter glass tube extending to within 13 mm from the bottom of the flask
Joints:	Inert, leak-free

An alternate method for cooling the stack gas is to use a condenser so designed that the total volume of condensate can be determined. A drying tube should follow the condenser to remove the remaining moisture for calculation of moisture content and to protect the vacuum pump and dry gas meter. Important specifications for the condenser and drying tube follow:

Condenser:	
Total capacity	1500 ml
Drying Column:	
Diameter	5 cm (2 in.)
Length	25 to 30 cm (10 to 12 in.)

2.1.6 Vacuum Pump

The vacuum pump must be capable of maintaining a constant flow rate of the sample gas. Two types of vacuum pumps are commonly used: a modified sliding fiber vane pump and a diaphragm pump. The pump should be leak-checked upon placing the pump in a sampling train such as in Figure 5-1 of Appendix A. With the pump running, adjust the system to 380 mm Hg (15 in. Hg) with the inlet of the metering system closed. The dry gas meter dial should not move after the adjustment has been made. When using a diaphragm pump with a by-pass valve, the absence of valve float must also be verified as directed in section 2.2.3. Suggested specifications for the vacuum pump follow:

Desired Flow Rate:	
At zero vacuum	0.11 m^3/min (4 ft^3/min)
At 508 mm Hg (20 in. Hg)	0.03 m^3/min (1 ft^3/min) (with the pump outlet at or near standard pressure, i.e., 760 mm Hg)
Vacuum (Inlet Plugged):	380 mm Hg (15 in. Hg)

2.1.7 Dry Gas Meter

A new dry gas meter must be calibrated according to section 2.2.3. It should meet the following requirement:

Accuracy:	±2 percent (compared to a wet test meter) at flow rates of .008 to 0.034 m^3/min (0.3 to 1.2 ft^3/min)

2.1.8 Orifice Meter

Construction details of the orifice meter are given by Martin (ref. 6). Significant design criteria are listed below. After visually inspecting the orifice meter and the inclined manometer (or equivalent differential pressure gage) for damage, the instruments are assembled in a sampling train as shown in Figure 5-1 of Appendix A and calibrated as directed in subsection 2.2.3. If ΔH@ is outside the limits outlined below (and discussed in subsection 2.2.3), the orifice meter should not be used with a commercially available sampling nomograph. It ΔH@ is greater than 53 mm (2.1 in.) of water, the orifice opening can be made larger (ref. 6) to lower ΔH@ to an acceptable value. Low values of ΔH@ cannot be corrected. Values of ΔH@ outside the limits listed below are acceptable when actual values are used to calculate the relationship of ΔH to ΔP (i.e., a nomograph is not used) for each field test. The value of ΔH@ should be inscribed on or otherwise affixed to the orifice meter box.

Specifications for this instrumentation include:

Orifice Diameter:	47 cm (3/16 in.)
ΔH@ at 21°C (70°F) and 760 mm Hg (29.92 in. Hg):	46.7 ± 6.4 mm H_2O (1.84 ± 0.25 in. H_2O)
Inclined Manometer Range:	0 to 200 or 250 mm H_2O (0 to 8 or 10 in. H_2O)
Inclined Portion of Scale:	
Range	0 to 25 mm H_2O (0 to 1 in. H_2O)
Divisions	0.25 mm H_2O (0.01 in. H_2O)
Vertical Portion of Scale:	
Range	25 to 200 or 250 mm H_2O (1 to 8 or 10 in. H_2O)
Divisions	2.5 mm H_2O (0.1 in. H_2O)

2.1.9 Thermometers

Three dial-type thermometers are suitable for monitoring the inlet and outlet temperatures of the dry gas meter and the sample gas as it leaves the last impinger. All of the above temperatures can be monitored by other means, such as a thermocouple or thermistor, but even then a dial-type thermometer is recommended as a backup system.

Dial-type thermometers are easily damaged. Each new thermometer should be visually checked for damage, such as a dented or bent stem. Each thermometer is then calibrated as directed in subsection 2.2.4. A thermometer should read within ± 3°C (± 5°F) of the true value when checked in an ice water bath and/or ± 4°C (± 7°F) when checked in boiling water (this represents a ± 1 percent accuracy when temperature is given in absolute units). Damaged thermometers that cannot be calibrated should be rejected.

Suggested thermometer data includes:

Range:	0 to 50°C (30° to 120°F)
Accuracy:	± 1% of absolute temperature

2.1.10 Vacuum Gage

The vacuum gage must be checked visually for damage and calibrated according to the directions in subsection 2.2.7. Any gage which is damaged and/or too erratic to be calibrated should be rejected.

A vacuum gage with the following specifications is adequate for monitoring the sampling train vacuum:

Range:	0 to 76 cm Hg (0 to 30 in. Hg)
Divisions:	25 mm Hg (1 in. Hg)

2.1.11 Check Valve

A one-way check valve equipped with convenient-sized fittings is required in the sampling train (see Figure 5-1 Appendix A).

2.1.12 Valves

Two metering valves (1 ball and 1 needle) with convenient-sized fittings are required in the sampling train. Locate the valves in the sampling train and check for proper operation. Reject any valve that cannot be adjusted over the desired operating range.

2.1.13 Stack Gas Velocity Measuring System

See the Quality Assurance Document of this series for Determination of Stack Gas Velocity and Volumetric Flow Rate (type-S pitot tube) (ref. 1) for a discussion of this system. Also, see the EPA revised method as contained in the Final Report of this contract (ref. 4).

2.1.14 Stack Gas Temperature Measuring System

This system is treated as a subsystem of the velocity measuring system and is discussed in the document referenced in the preceding subsection (i.e., 2.1.13). The temperature measuring system must be installed as shown in Figure 5-1 of Appendix A so that the sensor extends beyond the probe tip and does not touch metal. The position should be about 1.9 to 2.5 cm (0.75 to 1 in.) from the pitot tube and probe nozzle to avoid interference with the gas flow (ref. 7).

2.1.15 Stack Gas Pressure Measuring System

This system is treated as a subsystem of the velocity measuring system and is discussed in the document referenced in subsection 2.1.13.

2.1.16 Filter Media

The filter media are not reusable and all new filters must be visually checked for pinholes and tears. Any filter with a flaw is rejected. It is recommended that the surface alkalinity (pH) be measured for each lot of filters and that only filters meeting the specifications below be used. The pH of a filter can be checked in the following manner:

1. Randomly select seven filters out of every box of 100 filters.
2. Fold and place one of the filters into a 100 ml beaker. Add approximately 1 ml of deionized, distilled water for 4 cm^2 of filter material, bring to a boil, and hold for 1 minute. Make a slurry while the mixture is boiling by stirring the filter and water with a glass stirrer.

3. Cover the beaker and cool to room temperature. Measure the pH with a standardized pH meter.

4. Repeat Steps 2 and 3 above for the other six filters. If all seven pH values are between 6.0 and 8.0, accept the 93 remaining filters. If one or more pH values fall outside the above range, it is recommended that the remaining filters not be used.

The collection efficiency shall be determined as specified in section 3.1.1 of Appendix A. Test data from the manufacturer's quality control program should meet this requirement. Recommended purchasing criteria for the filters follow:

Material:	Glass-fiber; inert
Collection Efficiency:	99.95 percent ($\leq$ 0.05% penetration) for particulates larger than 0.3 μm in diameter
Surface Alkalinity:	pH = 7 ± 1

2.2 EQUIPMENT CALIBRATION

2.2.1 Nozzle Diameter

Upon the purchase of a new nozzle, measure the inside diameter to the nearest 0.025 mm (0.001 in.) using a micrometer. Make a minimum of three individual measurements using different diameters (rotate 60° for each measurement) each time and calculate an average diameter. The range (i.e., difference in smallest and largest values) of the measurements should not exceed 0.1 mm (0.004 in.). If it does, the nozzle is out-of-round and should be repaired or replaced. Number the nozzle and record the number and diameter of the nozzle to the nearest 0.025 mm (0.001 in.) in the laboratory calibration log book. Etch or permanently fix the average diameter on the nozzle. The nozzle should be checked visually for out-of-roundness and signs of damage before each field test.

2.2.2 Probe Heater and Filter Box Calibration

Set up the sampling train with the probe connected to the sampling box. With the nozzle detached and a flow rate of 0.021 m^3/min (0.75 ft^3/min), profile the temperature in the probe as the heat is increased. Determine the inlet and outlet temperature of the probe at a determined reference point corresponding to 120°C (248°F). If the outlet temperature is not 120°C ± 14°C (248 ± 25°F) and the temperature is reasonably uniform along the probe, adjustments must be made. If the probe is not equipped to monitor its temperature continuously, the temperature should be plotted as a function of the percent of the power setting (ref. 9).

A thermocouple with leads of approximately 3 meters can be used with a potentiometer readout to profile the probe temperature (draw the thermocouple along the length of the probe while maintaining a sample flow rate of 0.021 m^3/min). An alternate approach would be to use a remote reading thermometer.

The filter box temperature can be monitored with a thermocouple, thermister, or with a dial-type thermometer. Adjust the voltage supply until the box temperature is 120°C (248°F). The temperature should not vary more than ± 14°C (± 25°F) over a 2 hour period.

In all thermometer or temperature sensor calibrations the reference points should be an ice bath and boiling water.

The probe and filter box heating units should be calibrated when new, after repairs, or any time the presampling check indicates that the system is not functioning properly.

2.2.3 Dry Gas Meter and Orifice Meter

An initial check should be made of the sampling train to check for proper operation of the pump, dry gas meter, vacuum gage, and dry gas meter thermometer(s). After the metering system components have been checked, the vacuum system should be leak-checked. This is done by plugging the inlet side of the metering system, pulling a vacuum of 380 mm Hg (15 in. of Hg), and observing the dry gas meter. If the leakage exceeds .00057 m^3/min (0.02 ft^3/min), the leak(s) must be found and eliminated until the above specification is satisfied.

The dry gas meter should be calibrated when new, and a three-point check performed prior to each field trip. Calibration is performed by making simultaneous total volume measurements with a calibrated wet test meter (or equivalent volume measuring device) and the dry gas meter in the meter box. The wet test meter must be of the proper capacity and accuracy. For commercial sampling trains, the wet test meter capacity must be at least 0.03 m^3/rev (1 ft^3/rev) with an accuracy of ± 1 percent. The wet test meter must be of the proper capacity; otherwise, at the higher flow rates the linkage in the wet test meter will slip, resulting in erroneous volume measurements as recorded by the wet test meter. The recommended calibration procedures are as follows:

1. If the sampling train has a diaphragm pump and by-pass valve, the system must be checked for valve float. A one point check is specified in Section 5.3 of the reference method (Appendix A). A multipoint check covering the expected operating range is recommended here (ref. 8). (The wet test meter is not required while checking for valve float by this technique).

a) Initially, operate the system at 0.02 m^3/min (0.75 ft^3/min) for 10 minutes before taking data.

b) Operate the sampling system at orifice readings between 1.3 mm and 130 mm H_2O (0.05 and 5 in. H_2O).

c) Take data through the above range at flow rates of 0.002, 0.008, 0.014, 0.019, and 0.028 m^3/min (0.1, 0.3, 0.5, 0.7, and 1.0 ft^3/min), once with the by-pass valve closed (see Figure 5-1 Appendix A) and once with the by-pass valve completely open. Time each setting for 1 minute. Record the ΔH setting orifice meter reading and the initial and final volumes on the dry gas meter for each flow rate setting.

d) Calculate ΔH@ from the two sets of data (see ref. 8).

$$\Delta H@ = \frac{K \Delta H}{V^2} \qquad (1)$$

where $K = 4.48 \times 10^{-4}\ m^6$ in metric units, and
$K = 0.56\ ft^6$ in English units.

Plot two curves (one with the bypass valve opened and the other with it closed) of ΔH@ vs. the volume (V) recorded by the dry gas meter. If the valves are floating, the two curves will not coincide. Ideally, the curves should coincide and be horizontal over the whole range; in practice, the curve will probably have a slight slope.

This initial check for valve float should be performed (for trains utilizing a diaphragm pump and by-pass valve) once after building or purchasing a sampling train and must be repeated whenever a new pump is installed in the system. Valve float, if present, must be corrected (ref.8).

2. Determination of $\Delta H@$ and γ is as follows:

a) The wet test meter is placed upstream of the sampling system with its outlet connected to the inlet (sample umbilical connection) of the meter box (ref. 9). These connections must be leak-free.

b) Operate the pump at a flow rate near 0.02 m^3/min for 15 minutes to warm up the pump and wet the surface of the wet test meter.

c) Collect and record (as shown in Figure 2) the calibration data by setting ΔH on the orifice manometer and letting a given volume (V_w) of air pass through the wet test meter (the larger the volume, the greater the accuracy). Repeat the above procedures until the data are collected. Always have the by-pass valve open. A stop watch or laboratory timer is used to record the time (θ) required for V_w to pass through the wet test meter.

The symbols in Figure 2 are:

V_w = Gas volume passing through the wet test meter, m^3 (ft^3)

V_d = Gas volume passing through the dry gas meter, m^3 (ft^3)

t_w = Temperature of the gas in the wet test meter, °C (°F)

t_{d_i} = Temperature of the inlet gas of the dry gas meter, °C (°F)

t_{d_o} = Temperature of the outlet gas of the dry gas meter, °C (°F)

t_d = Average temperature of the gas in the dry gas meter, obtained by the average of t_{d_i} and t_{d_o}, °C (°F)

θ = Time of calibration run, minutes

ΔH = Orifice manometer setting, with a resultant orifice meter pressure drop, mm H_2O (in. H_2O)

γ = Ratio of volumetric measurement by wet test meter to dry test meter. Tolerance = $\pm$ 0.02

Date __________ Calibration By ________

Barometric Pressure, P_b = _______in.Hg

Dry Gas Meter No. ____________________

Wet Test Meter No. __________________

Wet Test Meter Capacity ______________

Date of Wet Test Meter Calibration __________________

Orifice manometer setting, ΔH, mm H_2O (in H_2O)	Gas vol. wet test meter V_w, $m^3(ft^3)$	Gas vol. dry gas meter V_d, $m^3(ft^3)$	Temperature				Time θ, min	Y	ΔH@
			Wet test Meter t_w, °C(°F)	Dry gas meter					
				Inlet t_{di}, °C(°F)	Outlet t_{do}, °C(°F)	Avg. t_d, °C(°F)			
2.5(0.1)	0.071(2.5)								
5.1(0.2)	0.071(2.5)								
7.6(0.3)	0.071(2.5)								
12.7(0.5)	0.14 (5)								
25.4(1.0)	0.14 (5)								
50.8(2.0)	0.28 (10)								
101.6(3.0)	0.28 (10)								
127.0(5.0)	0.28 (10)								
203.2(8.0)	0.28 (10)								
						Average			

Calculations

ΔH	$\frac{\Delta H}{13.6}$	Y $\frac{V_w P_b (t_d + 273)}{V_d\left(P_b + \frac{\Delta H}{13.6}\right)\left(t_w + 273\right)}$	ΔH@ $\frac{0.0012\,\Delta H}{P_b\,(t_{do} + 273)}\left[\frac{(t_w + 273)\,\theta}{V_w}\right]^2$
0.1	0.00735		
0.2	0.0147		
0.3	0.0221		
0.5	0.0368		
1.0	0.0735		
2.0	0.147		
3.0	0.221		
4.0	0.294		
8.0	0.588		

Figure 2. Dry gas meter and orifice meter calibration and calculation form.

P_b = Barometric pressure, mm Hg (in. Hg)

ΔH@ = Orifice meter pressure differential that gives a flow rate of 0.021 m^3/min (0.75 ft^3/min) at 21°C and 760 mm Hg, mm H_2O (in. H_2O). Tolerance = ± 3.8 mm H_2O (0.15 in. H_2O)

d) Calculate γ and ΔH@ for each orifice manometer setting and record on the calibration sheet as depicted in Figure 2. Plot curves of ΔH@ versus ΔH (orifice manometer setting in mm of water). The value of ΔH@ should be 46.7 ± 6.4 mm H_2O (1.84 ± 0.25 in. H_2O) with a variability no greater than ± 3.8 mm H_2O (0.15 in. H_2O) over a pressure differential range of 13 to 203 mm H_2O (0.5 to 8 in. H_2O) across the orifice. If this is not obtained, adjust the orifice opening or replace the orifice as directed in reference 6 and recalibrate. The value of γ should be 1.0 ± 0.02; adjust the linkage of the dry gas meter (if needed) as directed by the manufacturer until this tolerance is obtained or construct a calibration curve for the dry gas meter.

The value of ΔH@ obtained at a flow rate of 0.021 m^3/min should be etched on or permanently attached to the orifice meter. The completed form in Figure 2 is filed in the calibration logbook.

2.2.4 Thermometers

Thermometers are calibrated against a mercury-in-glass thermometer with at least 1°C divisions at two or three points as applicable. The points are an ice bath, room temperature, and boiling water (corrected for pressure). A thermometer should be calibrated when new and checked at one point in its expected operating range before each field test. A calibration curve should be constructed if the test thermometer does not read with ± 1 percent of the mercury-in-glass thermometer (both readings in °K).

Record all calibration data in the calibration logbook.

2.2.5 Stack Gas Velocity Measuring System

The procedure for calibrating a type-S pitot tube is given in the Quality Assurance Document of this series applicable to Method 2, Determination of Stack Gas Velocity and Volumetric Flow Rate (type-S Pitot Tube) (ref. 1), with one exception: the type-S pitot tube should be calibrated in the same configuration that it is to be used. For this method, the pitot tube is attached to the sampling probe. However, if a free space is maintained between the sampling nozzle and pitot tube of at least 1.9 cm (0.75 in.) (see Figure 5-1, Appendix A) during sampling, the pitot tube can be calibrated as directed in reference 1. If the sampling train is designed for sampling at higher than normal flow rates thereby requiring the use of larger size nozzles, the free space shall be set on the largest sized nozzle to be used. Method 2 as revised by EPA is contained in the Final Report of this contract (ref. 4).

The pitot tube coefficient should not vary more than ± 5 percent of the average over the operating range. If the average coefficient is outside the range of 0.83 to 0.87 and a sampling nomograph is used in maintaining isokinetic conditions, corrections must be made as directed in subsection 2.4.3.1.

2.2.6 Stack Gas Temperature Measuring System

A temperature-measuring device attachable to a pitot tube, as shown in Figure 5-1 of Appendix A, and capable of measuring the stack gas temperature to within ± 1.5 percent of the minimum absolute stack gas temperature is required. A high-quality mercury bulb thermometer calibrated at ice water and boiling water (corrected for local pressure) temperatures and readable to the nearest 0.5°C (1°F) is an acceptable laboratory standard for calibration of temperature-measuring devices. The calibration procedure is contained in section 2.1.2.2 of the Quality Assurance Document of this series for Method 2, Determination of Stack Gas Velocity and Volumetric Flow Rate (type-S Pitot Tube) (ref. 1).

2.2.7 Barometer

The field barometer should be checked against a mercury barometer before each field test. If the two differ by more than ± 5.1 mm (± 0.2 in.) of mercury either adjust, calibrate, or replace the field barometer as applicable. Record the results in the calibration logbook. Date and sign the entry.

In lieu of a field barometer the barometric pressure may be obtained from a nearby weather station. The uncorrected weather station pressure data, however, must be adjusted for elevation.

2.2.8 Analytical Balance

The balance calibration should be verified when first purchased by weighing a set of at least five standard weights covering the weight range from about 0.2 to 2 g. If any weighing deviates more than ± 0.3 mg from its stated value, the balance should be recalibrated. Subsequent checks are made by weighing a standard weight from the above weight range before each set of weighings of filters. The balance should be calibrated whenever a standard weight cannot be weighed to within ± 0.3 mg of its stated weight. The manufacturer or a qualified laboratory should perform the calibration.

2.2.9 Orsat Analyzer (optional)

A standard Orsat analyzer may be used at combustion sources for the determination of stack gas molecular weight. See the document of this series entitled "Gas Analysis for Carbon Dioxide, Excess Air and Dry Molecular Weight", based on Method 3 (ref.2).

2.3 PRESAMPLING PREPARATION

2.3.1 Preliminary Site Visit (optional)

The main purpose of a preliminary site visit is to gather information to design and implement an efficient source test. Prior preparation will result in the prevention of unwarranted loss of time, expenses, and injury to test and/or plant personnel. A test plan conceived from a comprehensive set of parameters will result in more precise and accurate results. This preliminary investigation (on-site) is optional and not a requirement. An experienced test group can, in most cases, obtain sufficient information on the source through communications with the plant engineer. The information should include pictures (diagrams) of the facilities.

2.3.1.1 Process (Background data on process and controls). It is recommended that the tester become familiar with the operation of the plant before a preliminary site visit is made. Data from similar operations that have been tested should be reviewed if they are available and applicable.

2.3.1.2 Sampling Site Preparedness. The management of each facility tested should provide an individual who understands the plant process and has the authority to make decisions concerning plant operation to work with the test team. This would include decisions concerning whether the plant would be operated at normal conditions or at rated capacity. This individual or individuals will supervise installation of ports, sampling platform, and electrical power. If the above installations are already in existence, they should be examined for their suitability for obtaining a valid test and for overall safety conditions (ref. 10). If the sampling platform, port size, and locations are sufficient, the diameter, area of the stack, and wall thickness should be determined. If ports have to be installed, specify at least 8 cm (3 in.) ports with plugs. Ten centimeter (4 in.) ports are preferred. Port locations should be based upon Method 1 of the Federal Register (ref. 11). One electric drop should be available at the test facility with 120-volt, 20-ampere service.

2.3.1.3 Stack Gas Conditions. The following can be determined on the initial site survey, either by measurement or estimation:

1. $T_{s_{avg}}$ = Approximate stack gas temperature
2. P_s = Static pressure (positive or negative)
3. ΔP_{max} and ΔP_{min} = Maximum and minimum velocity pressure heads
4. B_{wo} = Approximate moisture content
5. M_s = Molecular weight calculated from approximate gas constituent concentrations.

The above parameters can be roughly determined using an inclined manometer with a range of 0-127 mm H_2O (0-5 in. H_2O), a type-S pitot tube, and a manual thermometer or thermocouple attached to the pitot tube with a potentiometric readout device. The moisture content (approximate) can be determined by the wet-bulb dry-bulb method where acid gases are not present and at stack gas temperatures less than 360°K, Method 4 can be used if the wet-bulb dry-bulb method is not applicable, and the gaseous constituents by hand-held indicator kits. Nomographs are useful in checking and estimating the preliminary required data (ref. 12).

2.3.1.4 Methods and Equipment for Transporting Apparatus to Test Site. Ropes, block and tackle, and other hoisting equipment should belong in the repertoire of any stack sampler. The initial site visit should include a preconceived plan between plant personnel and tester on how the equipment can best be transported to the sampling site. Electric forklifts should be utilized when at all possible. In addition to the above, it is recommended, when permissible, that pictures be taken of the hoisting and sampling areas, so that any further correspondence (either by letter or telephone) will be clarified.

2.3.2 Apparatus Check

Each item to be used should be visually checked for damage and/or excessive wear before each field test. Items should be repaired or replaced if judged to be unsuitable for use by the visual inspection.

Figure 3 shows a checklist for the three phases of a field test. It is meant to serve as an aid to the individuals concerned with procuring and checking the required equipment, and as a means for readily determining the equipment status at any point in time. The completed form should be dated, signed by the field crew supervisor, and filed in the operational logbook upon completion of a field test. This includes initiating the replacement of worn or damaged items of equipment. Procedures for performing the checks are given in the appropriate subsections of this operations manual; a check is placed in the proper row and column of the checklist as the check/operation is completed.

PRESAMPLING CHECKLIST

TEST SITE____________________ CREW SUPERVISOR______________________ DATE____________ ___

Date and Initial Appropriate Block as Procedure is Completed

ITEM	Visual Check for Damage	Leak Check Sampling Train	Performance/ Calib. Check	Packed for Shipment
SAMPLING PREPARATION AND SAMPLING APPARATUS: 1. Nozzle				
2. Probe				
3. Filter Holder				
4. Filter Holder Box				
5. Filters				
6. Thermometers				
7. Impingers				
8. Vacuum Pump				
9. Dry Gas Meter				
10. Orifice Meter				
11. Inclined Manometer				
12. Vacuum Gage				
13. Check Valve				
14. Metering Valves				
15. By-Pass Valve				
16. Type-S Pitot Tube		▨		
17. Connecting Lines		▨		
18. Barometer		▨		
19. Inclined Manometer		▨		
20. Stack Temperature Measuring System		▨	▨	
SAMPLE RECOVERY: 21. Probe Brush		▨	▨	
22. Storage Containers		▨	▨	
23. Graduated Cylinders		▨	▨	
24. Wash Bottles		▨	▨	
REAGENTS: 25. Silica Gel		▨	▨	
26. Distilled Water		▨	▨	▨
27. Crushed Ice		▨	▨	▨
28. Filters		▨	▨	
DOCUMENTATION: 29. Data Sheets		▨		
CALCULATIONS & DATA VALIDATION: 30. Sampling Nomograph		▨		
31. Combustion Nomographs		▨		
32. Pocket Calculator		▨		
TOOLS AND EQUIPMENT: 33. Transportation Equipment		▨		
34. Safety Equipment		▨		
35. Tools and Spare Parts		▨		
36. Miscellaneous Supplies		▨		

Figure 3. Particulate measurement checklist

ON-SITE MEASUREMENT CHECKLIST

TEST ____________ CREW SUPERVISOR ____________ DATE ________

Date and Initial Appropriate Block as Procedure is Completed

ITEM	Unpacked or Purchased On-Site	Assembled and Performance Checked	Dissassembled and Packaged for Shipment
SAMPLING PREPARATION AND SAMPLING APPARATUS: 1. Nozzle		LEAK CHECK SAMPLING TRAIN	
2. Probe			
3. Filter Holder			
4. Filter Holder Box			
5. Filters			
6. Thermometers			
7. Impingers			
8. Vacuum Pump			
9. Dry Gas Meter			
10. Orifice Meter			
11. Inclined Manometer			
12. Vacuum Gage			
13. Check Valve			
14. Metering Valves			
15. By-Pass Valve			
16. Type-S Pitot Tube			
17. Connecting Lines			
18. Barometer			
19. Inclined Manometer			
20. Stack Temperature Measuring System			
SAMPLE RECOVERY: 21. Probe Brush			
22. Storage Containers		▨	
23. Graduated Cylinders		▨	
24. Wash Bottles		▨	
REAGENTS: 25. Silica Gel		▨	
26. Distilled Water		▨	▨
27. Crushed Ice		▨	▨
28. Filters		▨	
DOCUMENTATION: 29. Data Sheets		▨	
CALCULATIONS & DATA VALIDATION: 30. Sampling Nomograph		▨	
31. Combustion Nomographs		▨	
32. Pocket Calculator		▨	
TOOLS AND EQUIPMENT: 33. Transportation Equipment		▨	
34. Safety Equipment		▨	
35. Tools and Spare Parts		▨	
36. Miscellaneous Supplies		▨	

Figure 3. Particulate measurement checklist (continued)

POST-SAMPLING CHECKLIST

TEST SITE_____________________ CREW SUPERVISOR_________________________ DATE____________

Date and Initial Appropriate Block as Procedure is Completed

ITEM	Inspect for Damage and/or Excess Wear	Accepted for Future Use	To Be Replaced
SAMPLING PREPARATION AND SAMPLING APPARATUS: 1. Nozzle			
2. Probe			
3. Filter Holder			
4. Filter Holder Box			
5. Filters			
6. Thermometers			
7. Impingers			
8. Vacuum Pump			
9. Dry Gas Meter			
10. Orifice Meter			
11. Inclined Manometer			
12. Vacuum Gage			
13. Check Valve			
14. Metering Valves			
15. By-Pass Valve			
16. Type-S Pitot Tube			
17. Connecting Lines			
18. Barometer			
19. Inclined Manometer			
20. Stack Temperature Measuring System			
SAMPLE RECOVERY: 21. Probe Brush			
22. Storage Containers			
23. Graduated Cylinders			
24. Wash Bottles			
REAGENTS: 25. Silica Gel	▨		
26. Distilled Water	▨		▨
27. Crushed Ice	▨	▨	▨
28. Filters	▨	▨	
DOCUMENTATION: 29. Data Sheets			
CALCULATIONS AND DATA VALIDATION: 30. Sampling Nomograph			
31. Combustion Nomographs			
32. Pocket Calculator			
TOOLS AND EQUIPMENT: 33. Transportation Equipment			
34. Safety Equipment			
35. Tools and Spare Parts			
36. Miscellaneous Supplies			

Figure 3. Particulate measurement checklist (continued)

In addition to a visual check, the following performance and/or calibration checks are performed before each field test.

2.3.2.1 Sampling Train. The design specifications of the particulate sampling train used by EPA are described in APTD-0581 (ref. 6). Commercial models of this train are available. Each individual train must be examined to see if it is in compliance with the specifications in APTD-0581 (ref. 6) or its equivalent. In addition, the Office of Air Programs Publication No. APTD-0576 is a valuable source of information (ref. 9).

1. Nozzle. The nozzle is visually checked for damage, especially the sharp leading edge, and the tip opening is checked for out-of-roundness. If there is any sign of damage or out-of-roundness of the tip, the nozzle diameter should be calibrated according to subsection 2.2.1. Clean the nozzle by scrubbing with tap water, followed with a wash (2 hours) in 1:1 (V/V) hydrochloric acid-water wash and rinsed with distilled, deionized water. An alternate cleaning procedure when rust and/or organic materials are present is to precede the tap water wash by soaking the nozzle in sulfuric acid. At all times protect the knife edge from being damaged. A damaged knife edge must be repaired or the nozzle discarded. Minor repairs may be done by the tester with a file and assorted tools. An alternate approach, especially with major damage, is to send the nozzle to a qualified machine shop.

2. Probe. Check the probe in the following manner:

a) Disassemble probe and check for breakage of inner liner or damage to other parts of probe.

b) Clean all metal parts with acetone.

c) Reassemble probe and clean inner liner with brush, using tap water and acetone. In extreme cases, the glass liner can be cleaned with a stronger cleansing agent. Following the initial wash, the glass liner (interior surface) should be soaked for 2 hours in a 1:1 (V/V) hydrochloric acid-water wash. The final rinse should be with distilled, deionized water and acetone (reagent grade). The acid wash is easily accomplished by placing a glass female socket over the end of the male outlet of the probe. The acid is added to the inlet side, and the probe is allowed to stand vertically for the duration of the wash period.

d) Check to see if probe will heat to the required temperature of 120°C (248°F) to prevent condensation. The probe temperature can be profiled with a remote reading thermometer or with a thermocouple with a readout device.

e) The probe should be sealed on the nozzle side and checked for leaks at a vacuum of 380 mm Hg (15 in. Hg).

f) Cover the open ends of the probe with serum caps or equivalent.

At temperatures greater than 260°C (500°F) or if asbestos string has been used as a gasket between the glass probe and the union holding the nozzle to the probe and probe sheath, the probability of leakage exists. Most stacks have a negative pressure; therefore, a leak would introduce diluent air into the system and result in a low bias. This problem can be eliminated by:

a) Sealing the sheath from the outside air with a rubber stopper or its equivalent (ref. 6), and

b) Drilling a 0.3 mm (1/8 in.) hole in the sheath on the opposite side of the pitot tube just behind the nut.

This modification also prevents "out" gases resulting from deterioration of the probe from contaminating the stack sample (ref. 13).

3. Sampling Train Leak-Check. Assemble the sampling train as shown in Figure 2. With all the impingers empty, leak-check the sampling train by plugging the probe inlet and pulling a vacuum of 280 mm Hg (15 in. Hg). Leaks greater than 2 percent of the sampling rate (i.e., about 0.0006 m^3/min or 0.02 ft^3/min) as indicated by the dry gas meter should be found and corrected before continuing.

Note 1: The leak-check cannot be made through the probe if asbestos string is used in the gasket. In this case, leak-check as described in 2.4.3.6. Following this initial leak-check at 280 mm (15 in.) Hg vacuum, connect the probe and leak-check at 25 mm (1.0 in.) Hg vacuum. A leakage rate greater than 0.00057 m^3/min (0.02 ft^3/min) is unacceptable.

Note 2: If using stopcock grease, use only the high temperature type.

2.3.2.2 Dry Gas Meter Calibration Check. After the sampling train leak-check has been satisfactorily completed in the system as directed, follow the same procedure as used in calibrating the dry gas meter (see subsec. 2.2.3); make runs at ΔH settings equivalent to flow rates of about 0.01, 0.02, and 0.03 m^3/min (0.50, 0.75, and 1.0 ft^3/min). Calculate γ for each run (see equation in Fig. 2). If γ at either one of the three points falls outside the range of 1.0 ± 0.02, the dry gas meter should be (1) adjusted and recalibrated, (2) recalibrated and a calibration curve constructed, or (3) replaced. Record the results in the calibration log-book. Date and sign the entry.

2.3.2.3 Needle Valve(s) Check. The needle valve(s) should be disassembled and cleaned or replaced at any sign of erratic flow-rate behavior attributable to the needle valve as observed during the above checks or when unable to regulate the flow rate at desired levels. Document the adequacy of the needle valve with a check mark in the performance check column of the presampling checklist (Figure 3).

2.3.2.4 Probe Heater Check. Connect the probe heating system. The probe should become uniformly hot to the touch within a few minutes after being turned on. If it does not heat properly, repair or replace as necessary. Document as part of the sampling probe performance check for the presampling phase (Figure 3).

2.3.2.5 Filter Holder Box Heater. Check the heating system to verify that a temperature of 120 ± 14°C (248° ± 25°F), can be maintained for at least 1 hour at laboratory conditions. The maintenance of this temperature may be required in the field to prevent condensation.

2.3.2.6 Barometer. The barometer is checked as a part of the stack gas pressure measuring system in the Quality Assurance Document of this series applicable to Method 2 (ref. 1).

2.3.2.7 Stack Gas Velocity Measuring System. Check the velocity measuring system according to the directions given in the Quality Assurance

Document of this series for Method 2 and in the EPA revised Method 2 as contained in the Final Report of this contract (ref. 4). Visual and performance checks are documented in Figure 3 under visual check for damage and performance and/or calibration check for the presampling phase of the field test. If a calibration check is made, it should be recorded, dated, and signed in the calibration logbook.

2.3.2.8 Filter Media. The filter should be permanently numbered for identification on its backside and along the outer edge where particulates will not be collected. It should then be conditioned 24 hours in a dessiccator using anhydrous calcium sulfate at 20° ± 6°C (68° ± 10°F). Following the drying procedure, the filter is weighed on a calibrated analytical balance (see below) to the nearest 0.1 mg. A filter is considered to be at a constant weight when two consecutive weighings separated by at least 6 hours of dissication result in a difference in weight no greater than 0.5 mg.

than 0.5 mg.

Prior to the filter weighing, the balance calibration should be checked by weighing a standard weight between 0.2 and 2 g. Results of the check are recorded in the laboratory logbook with the filter weights. If the standard weight cannot be weighed within ± 0.3 mg of its stated value, the balance should be scheduled for calibration and a correction factor determined for the present weighings by weighing a set of standard weights spanning the range of interest.

The filter numbers and tare weights are recorded in the laboratory logbook. Each filter is placed in an inert container and sealed with the filter number written on the container.

2.3.3 Package Equipment for Shipment

An important aspect of *any* source testing method in terms of logistics, time of sampling, and quality of data is the packing of equipment with regard to (1) accessibility in the field, (2) ease of movement on site, and (3) optimum functioning of measurement devices in the field. Equipment should be packed under the assumption that it will receive severe treatment during shipping and field operation.

2.3.3.1 Type-S Pitot Tube and Probe. Pack the pitot tube and probe in a case protected by expanded polyethylene or other suitable packing material. An ideal container is a wooden case or equivalent lined with expanded polyethylene in which separate compartments are cut to hold the individual devices. It is also recommended that inserts for the individual nozzles be provided. The case should have handles that can withstand hoisting and should be rigid enough to prevent bending or twisting of the devices during shipping and handling.

2.3.3.2 Differential Pressure Gage (Dual Inclined Manometer). Always close all valves on the pressure gage. Pack it in a suitable case for shipment. Spare parts, such as O-rings and gage oil (dual inclined manometer), should also be packed.

2.3.3.3 Stack Temperature Measuring Device. The temperature-measuring device (thermocouple, thermistor, remote reading thermometer, etc.) should be protected from breakage; i.e., placed in a tube or a suitable shipping container. If the device is an integral part of the pitot tube, it can be shipped in the type-S pitot tube shipping case.

2.3.3.4 Barometer. The barometer should be packed in a shock-mounted (spring system) carrying case.

2.3.3.5 Pitot Tube Lines and Sample Line (Umbilical). All pitot tube lines and sample lines should be coiled to utilize the smallest amount of space. The ends should be connected together and sealed to prevent dust and dirt from impairing their operation. For shipment all lines should be stored in a case (foot locker) for protection and portability.

2.3.3.6 Glassware (Impingers, U-Joints, etc.). A word of caution is needed in the use of glassware. It is expensive and fragile, but with sensible handling and packing its failure rate and resultant costs are minimal. Generally, breakage of glassware occurs during packing and movement to the sampling facility. It is recommended that glass impingers be packed in a suitable case with approximate dimensions of 50 x 50 x 50 cm (20 x 20 x 20 in.) with a three-tiered layer of expanded polyethylene in which holes are cut to hold the glass impingers. At least 1.3 cm (1/2 in.) of cushioning material should be placed in both top and bottom of the shipping case (ref. 14). A separate case lined with expanded polyethylene, with layers of 8 cm (3 in.) polyethylene, can be used to carry the rest of the individual glass joints, filter holders, and filters. One major point to consider in shipping cases is the construction materials. Durable containers, although more expensive to build, are the most cost effective in the long term. A poorly constructed shipping case of inferior material will quickly deteriorate. Special packaging may be required if shipped by air freight.

2.3.3.7 Metering System (Meter Box Assembly). A standard (commerical unit) including pump, vacuum gage, dry gas meter, inclined manometer, etc., are contained in one meter box. This meter box should be placed in a shipping container lined with a cushioning material such as polyurethane.

If the vacuum pump is not integral to the meter box, it should be packed in a shipping container unless its housing is sufficient for travel. Additional pump oil should be packed with the pump if oil is required for its operation.

2.3.3.8 Sampling and Sample Recovery. Sampling and sample recovery equipment include the following:

1. Probe brush (commercial unit) or fabricated tube as long as the probe.
2. Glass wash bottles.
3. Glass storage containers. Chemically resistant, borosilicate narrow mouth glass bottles, 500 ml or 1000 ml for acetone washes. The caps for the containers should be lined with Teflon or some other inert material that is non-reactive to acetone.
4. Graduated cylinder (250 mℓ).
5. Silica gel: indicating type, 6-16 mesh, dried at 175°C (350°F) for 2 hours in preweighed lots of approximately 200 g (two/test plus spares in case of high moisture content in the stack gases).
6. Ice chest and water container.

7. Acetone: AA (atomic absorption) grade.
8. Distilled, deionized water.
9. Wash acid: 1:1 (V/V) hydrochloric acid-water solution.

All glass bottles or glass storage containers should be packed with cushioning material at the top and bottom of the case with some form of divider to separate the components. One shipping case can contain the acetone, preweighed silica gel, glass wash bottles, graduated cylinders, and probe brush. A water container and ice chest can be shipped as is. It is recommended in certain cases that these two items be purchased on-site. A general rule of thumb in source testing is "when possible, always carry a spare."

2.3.3.9 Source Sampling Tools and Equipment. The need for specific tools and equipment will vary from test to test. A listing of the most frequently used tools and equipment is given below:

1. Equipment Transportation

a) A lightweight handtruck that can be used to transport cases and that can be converted to a four-wheel cart for supporting the meter box control unit.

b) A 1.3 cm (1/2 in.) continuous filament nylon rope with large-throat snap hook and snatch block for raising and lowering equipment on stacks and roofs.

c) Tarpaulin or plastic to protect equipment in case of rain. Sash cord, 0.6 cm (1/4 in.) diameter for securing equipment and tarpaulin.

d) One canvas bucket, useful for transporting small items up and down the stack.

2. Safety Equipment

a) Safety harness with nylon and steel lanyards, large-throat snap hooks for use with lanyards for hooking over guard rails or safety line on stack.

b) A fail-safe climbing hook for use with climbing harness when climbing ladders having a safety cable.

c) Hard hats with chin straps and winter liners, gas masks, safety glasses or safety goggles, and a first aid kit.

d) Protective clothing, including the following: appropriate suits for both heat and cold, gloves (both asbestos and cloth), and steel-toed shoes.

e) Steel cable, .5 cm (3/16 in.) diameter, with thimbles, cable clips, and turn-buckles. These are required for installing a safety line or for securing equipment to the stack structure.

3. Tools and Spare Parts
   - a) Electrical and Power Equipment
     - 1) Circular saw
     - 2) Variable voltage transformer
     - 3) Variable speed electrical drill and bits
     - 4) Ammenter-voltmeter-ohmmeter (VOM)
     - 5) Extension cords: Light (No. 14 AWG), 2 cords x 8 m (25 ft) long
     - 6) 2-3 wire electrical adapters
     - 7) 3-wire electrical triple taps
     - 8) Thermocouple extension wire
     - 9) Thermocouple plugs
     - 10) Fuses
     - 11) Electrical wire
   - b) Tools
     - 1) Tool boxes (one large, one small)
     - 2) Screwdrivers
       - (a) One set flat blade
       - (b) One set Phillips
     - 3) C-clamps (2): 15 cm (6 in.), 8 cm (3 in.)
   - c) Wrenches
     - 1) Open end set: 0.6 to 2.5 cm (1/4 to 1 in.)
     - 2) Adjustables: 30 cm (12 in.), 15 cm (6 in.)
     - 3) One chain wrench
     - 4) One 30 cm (12 in.) pipe wrench
     - 5) One Allen wrench set

d) Miscellaneous

1) **Silicone sealer**
2) **Silicone vacuum grease (high temperature)**
3) **Pump oil**
4) **Manometers (gage oil)**
5) **Antiseize compound**
6) **Pipe fittings**
7) **Dry cell batteries**
8) **Flashlight**
9) **Valves**
10) **Dial thermometers, 150 mm (6 in.) and 915 mm (36 in.)**
11) **Vacuum gage**
12) **SS tubing: 0.6 cm (1/4 in.), 0.9 cm (3/8 in.), 1.3 cm (1/2 in.); short lengths**
13) **Heavy-duty wire (telephone type)**
14) **Adjustable packing gland**

2.3.3.10 Data Recording. Pack one large briefcase with at least the following:

1. **Nomograph for maintaining isokinetic conditions,**
2. **Data sheets or data notebook,**
3. **Carbon paper,**
4. **Slide rule or electronic calculator,**
5. **Psychrometric charts,**
6. **Combustion nomographs (ref. 12),**
7. **Pencils, pens, and**
8. **Calibration data, $\Delta H@$, γ, D_n, and C_p,**

2.4 ON-SITE MEASUREMENTS

The on-site measurement activities include transporting the equipment to the test site, unpacking and assembling the equipment, confirming duct measurements and traverse points (such preliminary determinations should be accomplished in a site visit), molecular weight determinations

of the stack gas, moisture content, setting of the nomograph, sampling, sampling recovery, and data recording. A sample data form is shown in Figure 4. Every quantitative stack or measurement data should be recorded on such a form.

2.4.1 Transport of Equipment to the Sampling Site

The most efficient means of transporting or moving the equipment from floor level to the sampling site (as decided during the preliminary site visit) should be used to place the equipment on-site. Care should be exercised against damaging the test equipment during the moving phase. Utilization of plant personnel or equipment (winches and forklifts), under close supervision, in moving the sampling gear is recommended.

2.4.2 Preliminary Measurements and Setup

2.4.2.1 Duct Measurement. Determine the number of traverse points according to Method 1 (EPA revised Method 1 in ref. 4). Measure the duct dimensions following the directions in subsection 2.2.3 of the Quality Assurance Document of this series for Method 2 (ref. 1).

2.4.2.2 Sample Box Logistics. Once the sampling points are selected and the probe has been marked with either a china marker or heat-bonding fiberglass tape (<370°C ≈ 700°F) the most efficient setup for the sample box must be determined. A poor choice will create a backbreaking and time-consuming sampling experience. Two rail systems exist for sampling, (monorails and duorails) in which the sampling box moves on a track(s). Each individual sampling situation will dictate the system of the sample box support. It is recommended that the sampling box be modified to allow at least two alternate methods of support.

2.4.2.3 Stack Gas Moisture Content. Determine the approximate moisture content of the stack gases by Method 4 or its equivalent (ref. 3). If the particular source has been tested before or a good estimate of the moisture if available, this should be sufficient. The reference method uses the condensate collected during the sampling for the moisture content used in final calculations.

TEST IDENTIFICATION

PLANT NAME ______________________ TEAM SUPERVISOR ________________
LOCATION ________________________ DATE OF TEST ____________________
SOURCE __________________________ TIME ____________
OPERATOR(S) _____________________

SAMPLING SITE IDENTIFICATION

STACK NUMBER ____________________
STACK DIMENSIONS:
d __________ m, L __________ m, W __________ m,
STACK AREA ____________________ m^2

APPARATUS IDENTIFICATION

NOZZLE DIAMETER __________ cm
DRY GAS METER NUMBER __________
γ __________
SAMPLING PROBE LENGTH __________
ORIFICE METER $\Delta H@$ __________ mm H_2O
PITOT TUBE NUMBER __________
PROBE LINER MATERIAL __________

DIFFERENTIAL PRESSURE GAGE:
RANGE __________ mm H_2O
DIVISION __________ mm H_2O
BAROMETER NUMBER __________
C_p __________

RUN INFORMATION

SAMPLE BOX NUMBER ________________ PROBE HEATER SETTING __________ °C
FILTER NUMBERS __________________ FILTER BOX HEATER SETTING ______ °C
SILICA GEL NUMBERS ________________
SAMPLING TRAIN LEAKAGE RATE ______ m^3/min at __________ mm Hg Vacuum

PRELIMINARY DATA FOR ISOKINETIC SAMPLING

ΔP_{avg} __________ mm H_2O
P_{bar} __________ mm Hg
P_g __________ mm Hg
$P_s = (P_{bar} + P_g)$ __________ mm Hg

B_{ws} __________
T_s __________ °K
"C" FACTOR __________

Figure 4: Sample Data Form for Particulate Emissions Determinations

RECORDED TEST DATA

TRAVERSE POINT NUMBER	SAMPLING TIME θ(MIN)	STACK TEMP. (°C)	VELOCITY HEAD ΔP (mm H_2O)	$\sqrt{\Delta P}$	ORIFICE METER ΔH(mm H_2O)	DRY GAS METER READING (m^3)	GAS SAMPLE VOLUME V_m (m^3)	DRY GAS METER TEMPERATURE		FILTER BOX TEMP (°C)	GAS TEMP. OF CONDENSER OR IMPINGER (°C)
								INLET (°C)	OUTLET (°C)		
TOTAL			▨			▨				▨	▨
AVERAGE			▨			▨	▨			▨	▨

Figure 4. Sample data form for particulate emissions determinations (continued).

MEASURED VALUES

(1) Total Particulate Weight Collected. m_n, mg.

CONTAINER NUMBER	WEIGHT OF PARTICULATE COLLECTED, mg		
	FINAL WEIGHT	TARE WEIGHT	WEIGHT GAIN
1			+ mg
2			+ mg
BLANK* CORRECTION			- mg
TOTAL (m_n)			mg

*Blank Correction = Residue (Blank) mg × (V_2/V_B)

where V_2 = Volume of acetone wash in container number 2, mℓ, and

V_B = Volume of acetone blank, usually 100 mℓ.

(2) Volume of gas sample through the dry gas meter at standard conditions to three significant digits.

$$V_{m(std)} = 0.3855\ V_m \left[\frac{P_{bar} + \frac{\Delta H}{13.6}}{T_m}\right] = \underline{\qquad\qquad}\ m^3.$$

(3) Moisture content of stack gas to two significant digits.

$$B_{ws} = \frac{(0.00134\ m^3/m\ell)V_{1_c}}{V_{m(std)} + (0.00134\ m^3/m\ell)V_{1_c}} = \underline{\qquad\qquad}\text{dimensionless.}$$

Figure 4. Sample data form for particulate emissions determinations (continued).

	VOLUME OF LIQUID WATER COLLECTED	
	IMPINGER VOLUME, ml	SILICA GEL WEIGHT, mg
FINAL		
INITIAL		
LIQUID COLLECTED		*
TOTAL VOLUME COLLECTED (V_{1_c})		mℓ

*Mass of water in mg = volume of water in mℓ.

(4) Particulate concentration at standard conditions on a dry basis.

$$C_s = (0.0154\ \text{g/mg})\,(m_n/V_{m(std)}) = \_\_\_\_\_\_\_\ \text{g/m}^3$$

(5) Stack gas molecular weight on a wet basis

$$M_s = M_d(1 - B_{ws}) + 18\ B_{ws} = \_\_\_\_\_\_\_\_\ \text{g/g-mole}$$

where $M_d = 0.44(\%\overline{CO}_2) + 0.32(\%\bar{O}_2) + 0.28(100 - \%\overline{CO}_2 - \%\bar{O}_2)$.

(6) Average stack gas velocity

$$(V_s)_{avg} = 34.97\ C_p\ (\sqrt{\Delta P})_{avg} \left(\frac{T_s}{P_s\ M_s}\right)^{1/2} = \_\_\_\_\_\_\ \text{m/sec}$$

(7) Percent of isokinetic sampling

$$I = \frac{4.323\ V_{m(std)}\ T_s}{\theta (V_s)_{avg}\ P_s\ A_n(1 - B_{ws})} = \_\_\_\_\_\_\_\_\_\_\ \text{percent.}$$

Figure 4. Sample data form for particulate emissions determinations (continued).

(8) Volumetric flow rate, dry basis, standard conditions

$$Q_s = 1388(1 - B_{ws})\,(V_s)_{avg}\,A(P_s/T_{s\ avg}) = \underline{\qquad\qquad}\ \text{m}^3/\text{hr.}$$

(9) Particulate mass emission rate calculated on a sample concentration basis

$$PMR = C_s \times Q_s = \underline{\qquad\qquad}\ \text{g/hr.}$$

SUMMARY OF RUNS

(10) Average particulate mass emission rate for three sample runs

$$\overline{PMR} = \frac{PMR_1 + PMR_2 + PMR_3}{3} = \underline{\qquad\qquad}\ \text{g/hr.}$$

(11) $\overline{PMR}$ with 90 percent confidence limits (for 3 runs) where $\overline{PMR}$ is the grand mean that would be obtained from a large number of tests

$$\overline{PMR} - 2.92\ s\{PMR\}/\sqrt{3} < \overline{PMR} < \overline{PMR} + 2.92\ s\{PMR\}/\sqrt{3}$$

$$\text{where } s\{PMR\} = \left[\frac{(PMR_1 - \overline{PMR})^2 + (PMR_2 - \overline{PMR})^2 + (PMR_3 - \overline{PMR})^2}{2}\right]^{1/2}$$

Figure 4. Sample data form for particulate emissions determinations (continued).

2.4.2.4 Molecular Weight of Stack Gas. Determine the dry molecular weight of the gas stream by Method 3 (ref. 2). If the particulate concentration is to be adjusted to 12% CO_2, it is recommended that the sample be of the integrated type for two reasons: 1) the possibility of a more representative sample and, 2) the convenience of taking the sample at the stack and being able to transport the sampling bag to a more suitable area for Orsat analysis.

2.4.2.5 Stack Temperature and Velocity Heads. Set up and level the dual inclined manometer and determine the minimum and maximum velocity head (ΔP) and the stack temperature (T_s). This is done most efficiently with a type-S pitot tube, with a temperature-sensing device attached as shown in Figure 5-1 of Appendix A. The ΔP's are determined with an inclined manometer by drawing the pitot tube across the stack diameter in two directions (circular stack with 90° traverses). This must be done in order to pick the correct nozzle size and to set the nomograph. Incorrect selection of nozzle size and/or setting of the nomograph may result in not being able to maintain the isokinetic sampling rate, thereby voiding the sample. Determine the static pressure as directed in the Quality Assurance Document of this series for Method 2 (ref. 1).

2.4.3 Sampling

The on-site sampling includes making a final selection of proper nozzle size, setting the nomograph (if used), loading the filter into the filter holder, preparing and assembling the sampling train, making an initial leak-check, inserting the probe into the stack, sealing the port, sampling isokinetically while traversing, recording the data, and making a final leak-check of the sampling system. Sampling is the foundation of source testing. Critical problems in testing result from poor or incorrect sampling more frequently than from any other part of the measurement process. The analytical process (laboratory) can never correct for errors made in the field resulting from poor judgment or instrumental failure. If the initial site survey, apparatus check and calibration, and preliminary measurement and setup on-site have been implemented properly, the testing should go smoothly with a minimal amount of effort and crises.

2.4.3.1 Preliminary Setting of the Nomograph. The setup of the nomograph using the parameters obtained in subsection 2.4.2 is given in detail in Appendix B of this document. A procedure is included in the appendix for checking the nomograph for correct design (accuracy).

Note 3: If the coefficient, C_p, of the type-S pitot tube being used is outside the range of 0.85 ± 0.02, compute the ratio $(C_p/0.85)^2$ and multiply this constant times the correction factor, C, obtained from the nomograph. Use this new "C" factor in setting the nomograph for isokinetic sampling (see Appendix B for further discussion).

2.4.3.2 Selection of Nozzle Size. After the nozzle size and appropriate probe length have been selected, insert the nozzle in the probe sheath union and tighten the union. Do not use wrenches; finger-tight is sufficient in most cases. Using uncontrolled pressure in tightening the union will result in a broken or cracked inner-liner. Keep the ball joint and nozzle tip protected from dust and dirt with a serum cap or equivalent.

2.4.3.3 Assembling of Sampling Train. Assemble the glass impinger train as follows:

1. Measure 200 mℓ of distilled water in the graduated cylinder 5-mℓ divisions) and place approximately 100 mℓ of the water in each of the first two impingers
2. The third impinger is left empty.
3. Place approximately 200 g of preweighed indicating silica gel into the fourth impinger.

The first, third, and fourth impingers are modified Greenburg-Smith while the second impinger is of the standard Greenburg-Smith design. Place the impingers into the sample box and assemble the sampling train using the appropriate U-joints. A very light coat of silicone grease (acetone insoluble) should be applied carefully to the joints, to avoid leaks in the system. Depending upon the design of the impinger, apply the lubricant in a manner that precludes its contact with the sample.

The loading of the impingers into the sample box can be done in the laboratory by sealing the inlet to the first impinger, the outlet of the third impinger, and inlet and outlet of the silica gel impinger. This is practical only when the sampling site is near and the logistics are suitable.

In view of the fact that the reference method does not consider the analysis of the condensible fraction, the impingers are not needed as such. A condenser can be employed consisting of a coiled tube of stainless steel or copper and a reservoir that has a capacity of at least 1500 mℓ. A good design would incorporate a drain valve in the bottom of the reservoir to allow the tester to drain and measure the total volume of condensate for moisture determinations. In all cases the impingers or condenser are maintained in an ice bath during sampling to remove the condensibles and keep the exit gas at or below 20°C (68°F). When a condenser is utilized, a drying tube downstream is used to protect the dry gas meter and vacuum pump.

2.4.3.4 Load Filter. Load the filter as follows:

1. Preweighed filter (desiccated for 24-hours and weighed to the nearest 0.1 mg) is removed with teflon covered tweezers from its sealed container and placed in the filter holder. The filter should have an identifying number and the filter holder should be numbered with a semi-permanent marker to preserve the integrity of the sample. Make certain that the filter is centered correctly in the holder with the sample side toward the probe. The filter holder should be tightened until the two halves are secure. Over-tightening the two halves can break the filter holder or tear the filter.

2. Place the filter holder into the sample box and connect the exit of the filter holder to the inlet of the first impinger. Plug the inlet of the filter holder with a glass ball to check for leaks. Connect the meter box (vacuum port) to the sample box with the appropriate umbilical cord.

2.4.3.5 Perform Leak-Check. Leak-check the sampling train by plugging the inlet to the filter holder, turning on the vacuum pump, and opening the valve system until the vacuum in the system reaches 380 mm Hg (15 in. Hg). A leakage rate not in excess of 0.0006 m^3/min (0.02 ft^3/min) at 380 mm Hg vacuum is acceptable. Release the pressure in the system but do not turn off the pump until the following sequence has been completed:

1. Slowly release the pressure in the system by carefully opening (twisting) the glass ball in the inlet of the filter holder.
2. Shut the coarse valve (main vacuum valve).
3. When the vacuum gage reads zero vacuum, remove the glass ball and shut down the pump.

2.4.3.6 Installation of Probe. Mount the probe in the sampling box. Check the configuration of the stack gas temperature measuring system as shown in Figure 5-1 in Appendix A. Connect the probe to the inlet of the filter holder and leak-check in the following manner:

1. Seal the inlet of the probe nozzle with a serum cap.
2. Turn on vacuum pump.
3. Open the valve system and adjust the vacuum to 380 mm Hg (15 in. Hg). A lower vacuum may be used provided that the vacuum used here is not exceeded during the test.
4. Check the leakage rate on the dry gas meter. A leakage rate less than 0.0006 m^3/min (0.02 ft^3/min) at 380 mm Hg (15 in. Hg) vacuum is acceptable.

Note 4: If an asbestos string is used in the fabrication of the probe nozzle to the probe liner connection, leak-check at 25 mm Hg (1 in. Hg) vacuum only. If a leak-free connection in the nozzle is employed, the total train, filter and probe, can be initially checked at 380 mm Hg (15 in. Hg) vacuum.

5. After completion of the leak check, release the pressure as follows:
   a) Slowly release the vacuum by carefully opening (squeezing) the serum cap until the system pressure is back to ambient (monitor with built-in vacuum gage).
   b) Turn valve system off (coarse valve).
   c) Turn off the vacuum pump.

Operations in subsections 2.4.3.5 and 2.4.3.6 can be combined, thereby requiring only one initial leak-check. Record leakage rate on the form in Figure 4.

2.4.3.7 Taking of Sample. Turn on the sample box and fill the impinger train container with crushed ice. The meter box operator should now recheck the setting of the nomograph while the sample box operator checks the filter box temperature gage to confirm that it is coming up to operating temperature; likewise, he can touch the probe to see if it is heating. It is recommended that a thermocouple be mounted next to the glass liner so that the probe temperature can be monitored.

As soon as the filter box temperature and probe temperature are up to the desired level, the test itself can be performed:

Remove the plug or cap from the sampling port and remove the dust (particulates) on the port walls by utilizing a wire brush or its equivalent. Remove the serum cap from the nozzle tip. Record the initial volume of the test meter on the data log sheet of Figure 4.

2. If the sample gas is hot, start at the traverse point farthest from the port and draw the probe out as the test continues. Asbestos gloves should be used in handling hot sampling probes and pitot tubes.

3. Attach a proper electrical ground to the probe and sampling system.

4. Insert the probe to the farthest traverse point with the nozzle pointing directly into the gas stream. Seal the port and immediately start the pump. Adjust the coarse and fine control valves until isokinetic conditions are obtained. Note the time and record it on the data log sheet of Figure 4.

5. Maintain isokinetic conditions during the entire sampling period. Sample for an equal amount of time at each traverse point. The time period at each traverse point should be long enough to set the sample rate and record the required data. The time period at each traverse point must be long enough to obtain a total sampling period representative of the process being monitored. The time at each traverse point must be sufficient to obtain a total sample volume of at least 1.7 m^3 (60 ft^3)

at standard conditions. While sampling, reset the nomograph if:

a) The temperature in the stack changes more than ± 14°C (25°F).

b) T_m (average temperature of meter) varies more than ± 6°C (11°F).

Adjust the sampling rate for every point and maintain the isokinetic rate by continuous observation. Record the meter volume when sampling has been completed at each individual traverse point. Take readings at each sampling point, at least every 5 minutes (or during sampling period at each traverse point): all the readings and adjustments should not be attempted for time intervals of less than 2 minutes. When significant changes in stack conditions are observed, compensating adjustments in flow rate should be made to maintain isokinetic conditions. Record on the data log sheet of Figure 4 the traverse point number, stack temperature (T_s), velocity pressure head (ΔP, mm H_2O or in. H_2O), (orifice pressure differential (ΔH, mm Hg or in. H_2O), gas temperature at dry gas meter ($T_{m_{in}}$) and $T_{m_{out}}$ or T_{avg}, °C or °F), sample box temperature, condenser temperature, and the probe temperature if the probe has a thermocouple and appropriate readout.

6. When sampling at one traverse point has been completed, move the sampler to the next point as quickly as possible. Close the control valve only when transferring the sampler from one sample port to the other. Exclude the time required to transfer the sampler from one port to another from the total sampling time.

Note 5: Since moving from port to port is time consuming, it is recommended that longer probes be employed to allow only one move during a test if a circular stack is involved. Probes up to 3 m (10 ft) long can be managed without too much difficulty, provided that adequate space is available on the sampling platform.

Upon transfer of the sampler to another port, the following procedures should be followed:

a) Monitor the vacuum through the system. An increase of vacuum is an indication of particulate buildup on the filter. Loss in vacuum is an indication of a broken impinger, connector, filter, or a loose connection.

b) Keep the impingers iced down (i.e., monitor the condenser temperature) to hold the temperature of the exit gas below 20°C (68°F). Add salt to the ice bath if necessary.

c) Check the line voltage with a voltmeter if a digital temperature system is utilized.

Note 6: Digital temperature systems may read erroneously with a drop in line voltage and/or interference from electromagnetic fields.

d) Make sure that the dual inclined manometer is level and that the pitot tube and pitot tube lines are unobstructed. A signal of trouble would be ΔP's that are not representative of the velocity heads obtained in the velocity traverse made during the preliminary site visit.

e) All data should be recorded on a data log sheet as depicted in Figure 4.

7. At the completion of the test, close the coarse control valve on the meter, remove the probe from the stack, and turn off the pump. Remove the probe carefully from the stack, making certain that the nozzle does not scrape dust from the inside of the port. Keep the nozzle elevated to prevent sample loss. After the probe cools place a serum cap or equivalent over the nozzle tip and leak-check the system at 50 mm Hg (2 in. Hg) vacuum above the operating vacuum during the test. The vacuum during this post sampling leak check should be no greater than 380 mm Hg (15 in. Hg). (Do not boil the water in the impingers.) Follow the same leak-check procedures as outlined in subsection 2.4.3.5. Seal the end of the nozzle. Disconnect the pitot tube lines and umbilical. Protect the pitot tube and the umbilical connections with tape or an appropriate equivalent. Record on the data log sheet of Figure 4 the leakage rate in m^3/min (ft^3/min) and the vacuum at which the leak check was performed. Check all connectors such as umbilical connection, pitot tube lines, glass connections, etc. for evidence of malfunction. Record all abnormalities on the data log sheet. The logging of abnormalities will not necessarily void the sample, but it may help to improve the quality of sampling performance.

2.4.4 Sample Recovery

Move the sampling train and probe to the sample recovery area. Care should be taken to prevent loss or contamination of the sample. If the probe must be removed before movement to the recovery area, the probe should be sealed at both ends (serum caps) and the inlet of the filter plugged with a glass ball. The sample recovery area should be a well-lighted, relatively clean room with enough table-top work space, about 4.6 m^2 (50 ft^2), for two crew members to change the filter and wash out the sampling train.

2.4.4.1 Container No. 1. Wipe the exterior of the filter holder surface to remove any excess dust or extraneous material. Remove the filter from the holder, place the filter in its original container and seal it. It is recommended that a piece of paper (inert, smooth surface) be placed under the filter holder as the filter is being removed to prevent loss of particulates. Removal of the filter is more efficient utilizing a set of tweezers. Teflon-tipped tweezers and a teflon scalpel should be used to handle filters. If a filter is torn, all pieces must be saved, conditioned, and weighed. Record date, time of test, location of test, and the number of run on this container. This data should also be recorded on the data log sheet of Figure 4.

2.4.4.2 Container No. 2. Wash all internal surfaces of the sampling train from the nozzle tip up to the backside of the filter holder with atomic absorption grade acetone. Determine the volume to the nearest ml and transfer to the container. A brush with a handle as long or longer than the probe should be used to loosen the particulate matter. It is recommended that the probe be washed by attaching a calibrated (125 mℓ or larger) cyclone flask to the end of the probe and washing the probe contents into the container. This wash may require emptying the flask into container No. 2 several times (the flask should be filled to the mark or to the nearest major division with acetone each time so that the quantity of wash transferred is accurately known). Also, a graduated cylinder should be used to accurately measure volumes of acetone (10 to 25 mℓ) to be used for rinsing the residue from the flask.

Accurately measure in a graduated cylinder an appropriate volume (e.g., 200 mℓ) of acetone to serve as a blank.

The measured volume of acetone wash is quite critical if the acetone is not residue free. The total volume of wash obtained by summing the volumes transferred to container No. 2 is recorded to the nearest mℓ.

Record total volume of acetone wash, date, time of test, location and run number on the container and on the data sheet of figure 4.

2.4.4.3 Container No. 3. Transfer the silica gel from the fourth impinger into its original preweighed container. Label container with date, time of test, location, and any other pertinent data. This information should also be recorded on the data log sheet. All sample containers should be glass with caps lined with teflon.

2.4.5 Total Volume

Measure the total volume of condensate by transferring the contents of the first three impingers into a graduated cylinder with divisions of 2 mℓ or less. Record this volume of condensate on the data log sheet of figure 4 to the nearest mℓ.

2.4.6 Sample Logistics (Data) and Packing of Equipment

The above procedures are followed until the required number of tests are completed. The following is recommended at the completion of testing.

1. Check all sample containers which must be properly labelled. (Time and date of test, location of testing, number of test, and any other pertinent documentation.) This aspect should be performed at the end of each individual test or prior to such test if the impingers are to be utilized in further tests before returning to the laboratory.

2. All data recorded during field testing should be recorded in duplicate (carbon paper). One set of data should be mailed to the base laboratory and the other to be hand-carried. This is a recommendation that can prevent a very costly mistake.

3. All sample containers should be properly packed in a sample box

for shipment to the base laboratory. All boxes should be properly labelled to prevent loss of the samples.

4. The sampling equipment should be inspected as it is disassembled for packing. Any signs of damage that could have had an influence on the precision/accuracy of the measurement should be documented on the data sheet and that item of equipment checked in the laboratory (if possible) to determine the magnitude of error that may have resulted from the damaged equipment.

2.4.7 Data Validation

Following the directions given in subsection 2.5.3, calculate and/or determine the following:

1. Moisture content of the stack gas, B_{ws}.
2. Stack gas molecular weight on a wet basis, M_s.
3. The average stack gas velocity, $(V_s)_{avg}$.
4. The percent of isokinetic sampling for the sample run, I.

Compare these measured values to theoretical values derived from combustion nomographs (ref. 12) or to values obtained by other measurement methods, e.g., measuring B_{ws} by the wet-bulb/dry-bulb method.

Any large unexplainable differences in measured and theoretical values should be noted and special care taken to reduce the variability of that specific parameter for the next run. If the percent of isokinetic sampling is outside the range of 0.90 to 1.10, the run should be repeated unless it is known (must have been approved by the administrator) that the particle size distribution is below, about 5 μm.

Values of B_{ws} and M_s as measured by the first run should be used in setting isokinetic conditions for subsequent runs unless there is reason to doubt their validity as compared to the values derived from preliminary measurements or estimates.

2.5 POSTSAMPLING OPERATIONS (Base Laboratory)

2.5.1 Apparatus Check

A postsampling check of the equipment can serve to validate the data from the just completed field test. The least it can do is aid the field team in making an honest estimate of the accuracy of the field measurements.

Any malfunctions uncovered during the postsampling check should be reported immediately and in detail to the supervisor. It should also be documented in the laboratory log and/or calibration log as applicable.

The decision of whether to correct the data, repeat the test, or just report the error with the data is one that the supervisor must make after considering 1) the magnitude of the error involved, 2) the precision/accuracy of the measurement process, and 3) the ultimate use of the field data.

2.5.1.1 Type-S Pitot Tube. The type-S pitot tube is checked according to the Quality Assurance Document of this series for Method 2 (ref. 1).

2.5.1.2 Dry Gas Meter and Orifice Meter (Sampling Train). A postcheck (a postcheck for one test can in some instances serve as the presampling check for the next test) should be made of the sampling train to check for proper operation of the pump, dry gas meter, vacuum gage, and dry gas meter thermometers. Leak-check the vacuum system. Determine γ and $\Delta H@$ at three points in the operating range. This is a check on the system for future testing and gives confidence in the data from the previous field test. This is a recommended procedure to improve the data quality and to prevent field sampling under assumed conditions.

2.5.2 Analysis (Laboratory)

The requirements for a precise and accurate analysis are minimal in the reference method. The analytical balance should be checked with a set of calibrated weights before the weighing of the first filter or at any time a problem is indicated. Record the actual and measured weights in the laboratory logbook along with the date and initials. If the actual and measured values agree to within ± 0.3 mg proceed; otherwise, report it to the supervisor before proceeding. Glass wash bottles should be utilized in transfer and the laboratory area should be free of any grinding or dust-producing activities. Blanks are required in the reference method and are always required in any analytical method striving for data validation.

2.5.2.1 Container No. 1. Transfer the filter and any loose particulate matter from the sample container to a tared glass weighing dish, desiccate and dry to a constant weight. Weigh and report results to the nearest 0.1 mg. After opening and closing, the humidity of the air in a desiccator may be very different from that of the atmosphere. Since the rate of removal of moisture by the desiccant may be quite slow, the initial period of desiccation should be at least 24 hours. After the initial period of desiccation, each succeeding period of desiccation should be at least 6 hours. Granular or fused anhydrous calcium sulfate is most frequently used as the drying agent. It has a good capacity, but is not a powerful desiccant, although sufficiently satisfactory in this respect to be usable in this work.

It is recommended that the sample be considered to be at constant weight when two successive readings separated by 6 hours vary by $\leq$ 0.5 mg. An analytical balance which weighs to 0.1 mg should be utilized.

2.5.2.2. Container No. 2. Transfer the (AA) acetone washings to a tared beaker and evaporate to dryness at ambient (laboratory) temperature and pressure. The transfer process should include washings (10 mℓ volumes premeasured in a graduated cylinder with 1 mℓ divisions) with (AA) acetone until a complete transferral (no signs of residual matter) is obtained. The total volume of these washings must be added to the wash volume obtained in the field (subsection 2.4.4.2). An accurately measured blank (100 mℓ measured in a graduated cylinder) of the acetone taken in the field is run concurrently with the samples. This blank will account for any residue in the acetone or any other laboratory condition that would tend to affect the final weight of the samples. After evaporation, the samples and blanks (one blank for each field site) should be desiccated until a constant weight is obtained. The same procedure as outlined in section 2.3.2.1 should be adhered to. Report results to the nearest 0.1 mg.

2.5.2.3 Container No. 3. Weigh the spent silica gel to the nearest 0.5 g. A top loading (trip) balance is sufficient for this weighing. It is recommended that the silica gel be weighed in Container No. 3 before the field test and returned to Container No. 3 following the test. This negates the error involved in not getting complete transferral of the silica gel from the original container.

All data (weights, volume of condensate collected in the first three impingers) are recorded on permanent data sheets such as depicted in Figure 4.

2.5.3 Calculations

Calculation error due to procedure or mathematical mistakes can be a large component of total system error. Therefore, it is recommended that each set of calculations be repeated, starting with the raw field data, preferably by a team member other than the one that performed the original calculations. If a difference greater than the typical round-off error is observed, the calculations should be checked step by step until the source of error is found and corrected. If a computer program is used, the original data entry should be checked and if differences are observed, a new computer run made. A standardized computer program should be written to treat all raw field data. A computer program presently being used in EPA is included in the Final Report of this contract (ref. 4).

2.5.3.1 Weight of Particulate Collected. Particulate weight is the sum of the weight gains of container 1 (filter and loose particulate) and container 2 (acetone probe wash) minus a correction for the blank as shown in the table under item (1) of Figure 4. Record m_n in the table of Figure 4 to the nearest 0.5 mg.

2.5.3.2 Dry Gas Volume. The sample volume measured by the dry gas meter is corrected to standard conditions by

$$V_{m(std)} = .3855\left(\frac{{}^\circ K}{mm\ Hg}\right) V_m \left(\frac{P_{bar} + \frac{\Delta H}{13.6}}{T_m}\right)$$

where $V_{m(std)}$ = Volume of gas sample through the dry gas meter at standard conditions, m^3

V_m = Volume of gas sample through the dry gas meter at meter conditions, m^3

P_{bar} = Barometric pressure at the orifice meter, mm H_2O.

ΔH = Average pressure drop across the orifice meter obtained from the table of recorded measurements of figure 4, mm H_2O.

T_m = Average dry gas meter temperature in °C plus 273 obtained from the table of recorded measurements of figure 4, °K.

Record $V_{m(std)}$ to the nearest 0.003 m^3 (0.1 ft^3) as item (2) under measured values on the data sheet in Figure 4.

2.5.3.3 Moisture Content, B_{ws}. Using the value of V_{1_c} from the table under item (3) of Figure 4, calculate the moisture content by

$$B_{ws} = \frac{(0.0013\ m^3/m\ell)\ V_{1_c}}{V_{m(std)} + (0.0013\ m^3/m\ell)\ V_{1_c}} .$$

Record B_{ws} to the nearest 0.001 on the data sheet under item 3 of Figure 4.

2.5.3.4 Particulate Concentration at Standard Conditions on a Dry Basis, C_s. Calculate the particulate concentration as follows:

$$C_s = (0.001\ g/mg) \left(m_n \middle/ V_{m(std)} \right)$$

where C_s = Particulate concentration in grams per standard cubic meter on a dry basis.

Record C_s to four significant digits on the data sheet under item 4 of Figure 4.

2.5.3.5 Stack Gas Molecular Weight on a Wet Basis, M_s. Calculate the stack gas molecular weight by

$$M_s = M_d\ (1 - B_{ws}) + 18\ B_{ws}$$

where M_d is given by

$$M_d = 0.44(\%\overline{CO}_2) + 0.32(\%\bar{O}_2) + 0.28(100 - \%\overline{CO}_2 - \%\bar{O}_2)$$

and $\%\overline{CO}_2$ and $\%\bar{O}_2$ are the averages of percent CO_2 and O_2 determinations, repsectively, according to the Quality Assurance Document of this series for Method 3 and the EPA revised method as contained in the Final Report of this contract (ref. 4).

Record M_s to three significant digits, i.e., --.-, on the data sheet under item 5 of Figure 4.

2.5.3.6 Average Stack Gas Velocity. Calculate the average stack gas velocity, $(V_s)_{avg}$, in m/sec by

$$(V_s)_{avg} = 34.97\ C_p\ (\sqrt{\Delta P})_{avg} \left[\frac{(T_s)^{*}_{avg}}{P_s\ M_s}\right]^{1/2}$$

where $(V_s)_{avg}$ = Average stack gas velocity, m/sec.

C_p = Average pitot tube calibration coefficient over the velocity range being measured, dimensionless.

$(\sqrt{\Delta P})_{avg}$ = Average of the square roots of the velocity pressure heads, $(\text{mm } H_2O)^{1/2}$.

$(T_s)_{avg}$ = Average absolute stack gas temperature, °K.

P_s = Absolute stack gas pressure, mm Hg.

M_s = Molecular weight of stack gas on a wet basis, g/g-mole.

Record $(V_s)_{avg}$ to three significant digits on the data sheet under item 6 of Figure 4.

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*If T_s varies more than about 10 percent of the mean from point to point in the stack, the correct term to use is $(\sqrt{T_s})_{avg}$ rather than $\sqrt{(T_s)_{avg}}$.

2.5.3.7 Percent of Isokinetic Sampling, I. Calculate the percent of isokinetic sampling by

$$I = \frac{4.323\ V_{m(std)}\ T_s}{\theta(V_s)_{avg}\ P_s\ A_n(1-B_{ws})}$$

where I = Percent of isokinetic sampling.

B_{ws} = Percent moisture by volume in the stack gas, dimensionless.

$V_{m(std)}$ = Volume of gas sample through the dry gas meter at standard conditions, m^3.

θ = Total sampling time, min.

$(V_s)_{avg}$ = Average stack gas velocity calculated in section 2.5.3.6 above, m/sec.

P_s = Absolute stack gas pressure, mm Hg.

A_n = Cross-sectional area of the nozzle, m^2.

Record I to two significant digits on the data sheet under item 7 of Figure 4.

2.5.3.8 Volumetric Flow Rate at Standard Conditions on a Dry Basis, Q_s. Calculate the volumetric flow rate by

$$Q_s = 1388(1 - B_{ws})\ (V_s)_{avg}A\ (P_s/T_{s\ avg})$$

where A = Cross-sectional area of the stack, m^2, and the other terms are as defined in the above calculation of stack gas velocity, subsection 2.5.3.6. A standard absolute temperature of 293°K and a standard absolute pressure of 760 mm Hg were used in the calculation of the constant.

Record Q_s to three significant digits on the data sheet under item 8 of Figure 4.

2.5.3.9 Particulate Mass Emission Rate Calculated on a Sample Concentration Basis, PMR. Calculate the particulate matter emission rate by

$$PMR = C_s \times Q_s$$

where PMR = Particulate mass emission rate, g/hr.

C_s = Particulate concentration at standard conditions on a dry basis, g/m^3.

Q_s = Volumetric flow rate at standard conditions on a dry basis, m^3/hr.

Record PMR to three significant digits on the data sheet under item 9 of Figure 4.

2.5.3.10 Calculation Check. Calculation error due to procedure or mathematical mistakes can be a large component of total system error. Therefore, it is recommended that each set of calculations be repeated, starting with the raw field data, preferably by a team member other than the one that performed the original calculations. If a difference greater than the typical round-off error is observed, the calculations should be checked step by step until the source of error is found and corrected. If a computer program is used, the original data entry should be checked and if differences are observed, a new computer run made. A standardized computer program should be written to treat all raw field data.

SECTION III MANUAL FOR FIELD TEAM SUPERVISOR

3.0 GENERAL

The term "supervisor" as used in this document applies to the individual in charge of a field team. He is directly responsible for the validity and the quality of the field data collected by his team. He may be a member of an organization which performs source sampling under contract to government or industry, a government agency performing source sampling, or an industry performing its own source sampling activities.

It is the responsibility of the supervisor to identify sources of uncertainty or error in the measurement process for specified situations and, if possible, eliminate or minimize them by applying appropriate quality control procedures to assure that the data collected are of acceptable quality. Specific actions and operations required of the supervisor for a viable quality assurance program are summarized in the following listing.

1. Monitor/Control Data Quality

a. Direct the field team in performing field tests according to the procedures given in the Operations Manual.

b. Perform or qualify results of the quality control checks (i.e., assure that checks are valid).

c. Perform necessary calculations and compare quality control checks to suggested performance criteria.

d. Make corrections or alter operations when suggested performance criteria are exceeded.

e. Forward qualified data for additional internal review or to user.

2. Evaluate Routine Operation

a. Obtain from team members immediate reports of suspicious data or malfunctions. Initiate corrective action or, if necessary, specify special checks to determine the trouble; then take corrective action. Document the corrective action taken.

b. Examine the team's log books periodically for completeness and adherence to operating procedures.

c. Approve data sheets, calibration checks, etc., for filing.

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3. Evaluate Overall System

a. Evaluate available alternative(s) for accomplishing a given objective in light of experience and needs,

b. Evaluate personnel training/instructional needs for specific operations.

Consistent with the realization of the objectives of a quality assurance program as given in section I, this section provides the supervisor with brief guidelines and directions for:

1. Collection of information necessary for assessing data quality on an intrateam basis;

2. The use of performance criteria to insure the collection of data of acceptable precision/accuracy;

3. Isolation, evaluation, and monitoring of major components of system variability.

In subsection 3.1, a method of assessing data quality on an intrateam basis is given. This method involves calculating a sample standard deviation using the three replicate runs required in a field test and calculating 90 percent confidence limits for the average of the three replicates.

Subsection 3.2 presents suggested criteria for judging equipment performance, frequency of calibration, and isokinetic sampling.

Directions for the collection and analysis of information to identify trouble, and subsequently, the control of data quality within acceptable limits are given in the third subsection.

3.1 ASSESSMENT OF DATA

The particulate mass emission rate, $\overline{PMR}$, for a particular field test is the average of at least three replicates. Intrateam assessment of data quality as discussed herein provides for an estimate of the precision of the measurements. Precision in this case refers to replicability, i.e., the variability among replicates and is expressed as a standard deviation. This precision statement combines variability due to process changes and to random measurement errors, This technique does not provide the information necessary for estimating measurement bias (see subsection 4.1.2 for a discussion of bias) that could occur, for example, from an error in determining the pitot tube coefficient, nozzle cross-sectional area, or the orifice meter calibration. However, if the operating procedures given in the Operations Manual are followed, the bias should be small in most cases. An independent performance audit which would make possible a bias estimate is suggested and discussed in section IV, the Manual for Manager of Groups of Field Teams.

3.1.1 Calculating Precision of Field Data

Each field test is comprised of at least three sample runs. Using the sample runs as replicates, a standard deviation can be calculated. This calculated standard deviation is a combined measure of the measurement and process variabilities. The standard deviation is calculated by

$$s\{PMR\} = \left[\frac{(PMR_1 - \overline{PMR})^2 + (PMR_2 - \overline{PMR})^2 + (PMR_3 - \overline{PMR})^2}{2}\right]^{1/2}$$

where $s\{PMR\}$ = The calculated standard deviation for the three sample runs, g/hr,

$PMR_1(PMR_2)(PMR_3)$ = Particulate mass emission rate for sample run 1 (2)(3), g/hr,

$\overline{PMR}$ = Average particulate mass emission rate of the three sample runs, i.e., $1/3(PMR_1 + PMR_2 + PMR_3)$, g/hr, and

2 = The number of replicates minus one (degrees of freedom).

3.1.2 Reporting Data Quality

The average measured particulate mass emission rate ($\overline{PMR}$) serves as a point estimate of the true average particulate mass emission rate ($\overline{PMR}_t$). The spread in the PMR's from the three runs can be used to calculate an interval estimate of $\overline{PMR}_t$. The procedure used here assumes that the measured $\overline{PMR}$ is normally distributed about $\overline{PMR}_t$ (i.e., the measurement method is not biased) with a variance estimated by $s^2(PMR)/n$. Since there is no way of determining or knowing $\overline{PMR}_t$ the internal estimate is actually applicable to the measured grand mean value $\overline{\overline{PMR}}$ that would result if the number of runs (n) became very large. If, in fact, the measurement method is unbiased then $\overline{PMR}_t = \overline{\overline{PMR}}$ as n approaches infinity.

It is recommended that the average particulate mass emission rate, $\overline{PMR}$, from the three runs be reported along with 90 percent confidence limits for the grand mean, $\overline{\overline{PMR}}$. The average measured value and calculated standard deviation are used to calculate 90 percent confidence limits for $\overline{\overline{PMR}}$ by

$$\overline{PMR} \pm 2.92\, s(\overline{PMR})/\sqrt{n}$$

where $\overline{PMR}$ = The average of three replicates, g/hr.

$s(PMR)$ = Estimated standard deviation of PMR based on three replicates, g/hr.

2.92 = 95th percentile of the Student t-distribution with 2 degrees of freedom which yields a 90 percent confidence interval.

n = The number of replicates, i.e., n = 3 for this case.

For example, if for a given field test $\overline{PMR} = 22.86$ g/hr and $s(PMR)$ is calculated to be 1.32 g/hr, the reported value with 90 percent confidence limits would be

$$22.86 \text{ g/hr.} \pm (2.92)\ (1.32 \text{ g/hr.})/\sqrt{3}$$

or the grand mean particulate mass emission rate, $\overline{\overline{PMR}}$, would be assumed (with 90% confidence) to be in the interval.

$$20.6 \text{ g/hr.} \le \overline{\overline{PMR}} \le 25.1 \text{ g/hr.}$$

The utility of the above statement follows from the fact that if this procedure for computing confidence limits is followed for several field tests, then 90 percent of the time the grand mean $\overline{\overline{PMR}}$ value will be contained within the given limits. It is recommended that the 90 percent confidence limits be reported with the Field Data Form in the Operations Manual.

3.2 SUGGESTED PERFORMANCE CRITERIA

Data assessment as discussed in the previous subsection is based on the premise that all variables are controlled within a given level, thereby guarding against large undetected biases in the measurement process. These levels of suggested performance criteria are the values given in the Operations Manual for determining when equipment and/or personnel variability is out of control. Criteria for judging performance are summarized in Table 1.

The following section discusses important sources of error in Method 5 and provides information on techniques for monitoring these variables to determine if the performance criteria given in this section are being exceeded.

3.3 COLLECTION AND ANALYSIS OF INFORMATION TO IDENTIFY TROUBLE

In a quality assurance program, one of the most effective means of preventing trouble is to respond immediately to indications of suspicious data or equipment malfunctions. There are certain visual and operational checks that can be performed while the measurements are being made to help insure the collection of data of good quality. These checks are written as part of the routine operating procedures in section II. In order to effectively apply preventive-type maintenance procedures to the measurement process, the supervisor must know the important variables in the process, know how to monitor the critical variables, and know how to interpret the data obtained from monitoring operations. These subjects are discussed in the following subsections.

3.3.1 Identification of Important Variables

Determination of the particulate mass emission rate requires a sequence of operations and measurements that yields as an end result a number that serves to represent the average particulate mass emission rate for that field test. There is no way of knowing the accuracy, i.e., the

Table 1. Suggested performance criteria

1. Suggested Criteria for Equipment Performance

(a)	Dry Gas Meter:	$.98 \le \gamma \le 1.02$
(b)	Barometer:	± 5.1 mm Hg (± 0.2 in. Hg)
(c)	Thermometers:	± 2.3°K (± 5°R) at 273°K (492°R) or ± 4°K (± 7°R) at 373°K (672°R)
(d)	Stack Temperature Measuring System:	± 2.3°K (± 5°R) at 273°K (492°R) or ± 4°K (± 7°R) at 373°K (672°R)
(e)	Sampling Train Leakage:	Less than 0.0006 m^3/min (0.02 ft^3/min) at 380 mm Hg (15 in. Hg) vacuum
(f)	Sample Box Heating System:	Capable of maintaining a temperature of 120°C (248°F) ± 14°C (25°F) at laboratory conditions.
(g)	Probe Heating System:	Uniform heating of probe with a temperature of 120°C (248°F) at the exit end at a flow rate of 354 cm^3/sec (0.75 ft^3/min) at room temperature.
(h)	Meter Orifice:	$\Delta H@$ should be 46.7 ± 6.4 mm H_2O (1.84 ± 0.25 in. H_2O) and not vary more than ±3.8 mm H_2O (±0.15 in. H_2O) over the range of operation of 13 to 200 mm H_2O (0.5 to 8 in. H_2O), if a commercial nomograph is used during sampling to aid in maintaining an isokinetic sampling rate.
(i)	Probe Nozzle Diameter:	Range of three different diameter measurements less than .010 cm (0.004 in.).
(j)	Analytical Balance:	Weigh a standard weight within ± 0.3 mg of its stated weight.
(k)	Type-S Pitot Tube:	C_p constant within ± 5 percent over working range and each calibration check is within 1.2 percent of the original C_p.
(l)	Filter Media:	$6.0 \le pH \le 8.0$

2. Suggested Criteria for Performing Equipment Calibration

(a) Above items (a) through (j) are calibrated when new and checked before each field test and recalibrated any time the check results fall outside the prescribed performance limits.

(b) Item (k), the type-S pitot tube is calibrated when new, before every third field test, or at any sign of damage.

(c) Item (l), filter media, the pH's of a random sample of 7 out of 100 filters are measured for each new order of filters. The remaining 93 filters are accepted if all 7 pH's are in the interval of 6.0 to 8.0.

3. Suggested Criteria for Percent Isokinetic

$$0.90 \le I \le 1.10$$

agreement between the measured and the true value, for a given field test. However, a knowledge of the important variables and their characteristics allows for the application of quality control procedures to control the effect of each variable at a given level during the field test, thus providing a certain degree of confidence in the validity of the final result.

A functional analysis of this method of measuring the particulate mass emission rate of a stationary source was made (see subsection 4.1) to try to identify important components of system error and show how these errors propagate through the measurement process and influence the final result. Results of an evaluation study of Method 5 (ref. 15) for within-run variations showed an average coefficient of variation of about 6 percent for twelve runs, six of which had four sampling trains and the other six runs had three sampling trains. Also, collaborative tests of the method (refs. 16, 17, and 18) show within laboratory CVs ranging from 10 to 30 percent and between laboratory CVs of 20 to 40 percent. These results are used in the functional analysis as an estimate of overall system error, while the individual error components are estimated using engineering judgment in a manner such that their combined variability is consistent with overall system error.

Variability in emissions data derived from multiple repetitions include components of variation from:

1. Process conditions,
2. Equipment and personnel in field procedures, and
3. Equipment and personnel in the laboratory.

In many instances time variations in source output may be the most significant factor in the total variability. In order to judge the relative magnitudes of measurement variability and process output variability, process parameters should be monitored throughout the test. Process information is also required if the particulate mass emission rate is to be given as a function of fuel input. The exact process data to be obtained are dependent upon the process being tested. In general, all factors which have a bearing on the emissions should be recorded on approximately a 15-minute interval (ref. 19). These factors include process or fuel weight rate, production rate, temperature and pressure in the reactor and/or boiler, control equipment, fan and/or damper settings, pressure drop or other indicators of particulate collection efficiency and opacity of exit plume. Sample forms for combustion, incineration, and process sources are given in reference 19.

It is most important to realize that the larger measurement errors result from poor operator technique such as loss or gain of collected particulate mass during sample recovery, (ref. 17), or poor orientation and positioning of the probe during sample collection. Such deviations from recommended procedures cannot be evaluated or corrected for. It is important to observe and eliminate such occurrences while the test is in process.

Sources of variation due to equipment includes the type-S pitot tube coefficient, sampling nozzle cross-sectional area, orifice meter, dry gas meter, probe heater, non-neutral filter media, and sample box heater. These parameters are all controlled through performance of calibration checks before each field test, and in the case of the filter media, by an acceptance check for new batches of filters. Also, the probe and sample box heaters are checked periodically during the conduction of the test.

Important error sources checked immediately before and/or during sample collection includes sampling train leaks, the sample gas temperature leaving the last impinger, and isokinetic sampling conditions.

Assuming good operator technique, the error sources named in the last two paragraphs are discussed and each one's effect on the determination of the particulate mass emission rate is derived from a functional analysis of the measurement process in subsection 4.1.

A summary of the important parameters is given below. The parameters are roughly given in ascending order of importance. Importance is qualitatively derived from the estimated error range of a parameter weighted by its estimated frequency of occurrence.

3.3.1.1 Equipment Calibration. Equipment calibration is the backbone of any quality assurance program. It is important that the calibration procedure be carried out correctly, that the calibration standards are properly calibrated and maintained, and that the frequency of calibration is adequate.

Important calibration constants and how they influence measurement accuracy include the following:

1. Error in the pitot tube calibration coefficient is directly reflected in the stack gas velocity determination and is doubled in the process of determining isokinetic sampling rates. This error could be very large if the pitot tube is not calibrated under actual field test conditions, i.e., strapped to the sampling probe and the spacings shown in Figure 5-1 of Appendix A are not maintained (ref. 7). Errors as large as 20 percent have been attributed to the pitobe configuration (ref. 7).

2. Error in determining the average nozzle diameter is quadrupled in the process of determining isokinetic sampling rates and is doubled in the percent of isokinetic sampling calculation. One source of error here is the use of an out-of-round nozzle. The average nozzle diameter is used to calculate the area of a circle which yields a larger than true cross-sectional area if the nozzle tip is not round.

3. Dry gas meter inaccuracy appears directly in the concentration and particulate mass emission rate determinations.

4. The orifice meter calibration constant is used in determining isokinetic sampling conditions and any error in the constant is doubled in setting the sampling rate. Also, if ΔH@, the pressure drop across the orifice that gives a flow rate of 0.21 m^3/min (0.75 ft^3/min) at 21°C (70°F) and 760 mm Hg (29.92 in. Hg) differs from 46.7 mm H_2O (1.84 in. H_2O), and a nomograph is used to set isokinetic sampling conditions, an error results. It is recommended that an orifice meter with a ΔH@ outside the range of 40.4 to 53.1 mm H_2O (1.59 to 2.09 in. H_2O) not be used in conjunction with a nomograph.

3.3.1.2 Anisokinetic Sampling. Anisokinetic sampling can occur from error in the calibration constants of the pitot tube, orifice meter, and nozzle diameter. It can also result to a lesser degree, usually, from measurement error in the moisture content and molecular weight of the stack gas. Errors from the above sources will not be directly reflected in the percent of isokinetic sampling calculation. Therefore, it is important to determine each parameter as accurately as possible, either through calibrations or careful measurements.

Failure, or in some instances the inability, to make adjustments in the sampling rate as the stack gas velocity varies or as the deposited particulate matter plugs the filter can result in anisokinetic sampling. Use of a nomograph can be a cause of anisokinetic sampling because of (1) any inaccuracy in the nomograph, (2) use of preset values for C_p, $\Delta H@$, and M_d (these errors can be eliminated by using actual values and adjusting the correction factor on the nomograph), and (3) operator error in setting the nomograph. The sum of these errors is quantified to a certain extent by the percent of isokinetic sampling calculation.

Deviation from isokinetic sampling cannot be related directly to error in the measurement process (see subsection 4.1). However, failure to maintain isokinetic sampling conditions under otherwise normal operations reflects the lack of alertness and, perhaps even the level of competency, of the field crew.

3.3.1.3 Sample Recovery. The technique used by, and the attitude of, the crew members in sample recovery are of paramount importance to measurement precision and accuracy (refs. 17 and 18). Use of an adequate sample recovery area in terms of space, lighting, or cleanliness will decrease the probability of error. Sample recovery procedures as given in Section 4.2 of Appendix A should be followed by the field team.

3.3.1.4 Calculations. Calculations for this method are known to be a major source of error (ref. 18). Some calculations involve several terms and should only be attempted (for the final report) at a desk or work table and preferably with the aid of a calculator or at least a good slide rule. A computer program using raw data as an input is highly recommended for making the final calculations.

As a check, it is recommended that all calculations be independently repeated from raw data.

3.3.1.5 Filter Media. This is included as a possibility. It is not known how important the pH of the filter media is at this time. However, data referenced in subsection 4.1 indicates that error due to the conversion of acid gases to particulate matter by alkaline filters could be significant. This error would be a positive bias resulting in a measured emission rate greater than the true rate.

3.3.2 How to Monitor Important Variables

In general, if the procedures outlined in the Operations Manual are followed, the major sources of measurement variability will be in control. It is felt, however, that the supervisor should visually check certain parameters and operations periodically while measurements are being made to insure good operator technique and the proper use of equipment. The parameters and operations to check are essentially those recommended for the auditor as listed in table 7 of subsection 4.3.

Results of the calibration checks for the dry gas meter, orifice meter, nozzle diameter, and pitot tube should be checked before each field test. Any item of equipment not satisfying the suggested performance criteria of table 2 should be calibrated or replaced.

There appears to be a need for actual field data on several of the parameters or variables involved in this measurement method in order to better judge their influence on measurement variability. One of the most effective means of identifying and quantifying important sources of variability is through the use of quality control charts. Quality control charts will provide a basis for action with regard to the measurement process; namely, whether the process is satisfactory and should be left alone, or the process is out of control and action should be taken to find and eliminate the causes of excess variability. In the case of this method in which documented precision data are scarce, the quality control charts can be evaluated after a period of time to determine the range of variation that can be expected under normal operating conditions. Also, even though results from individual field tests are within bounds, trends can be identified and corrective action taken further improving data quality through the proper use of control charts.

Discussions of control charts and instructions for constructing and maintaining them are given in many textbooks in statistics and quality control, such as in references 20 and 21. Also, volume 1 of the Quality Assurance Handbook for Air Pollution Measurement Systems published by EPA discusses the use of control charts (ref. 22).

It is good practice to note directly on control charts the reason for out-of-control conditions, if determined, and the corrective actions taken. Recommended control charts are discussed below.

3.3.2.1 Pitot Tube Calibration Coefficient. A sample control chart for pitot tube calibration checks is given in the quality assurance document of this series for Method 2 (ref. 1).

3.3.2.2 Determination of %CO_2 with Orsat Analyzer. For incinerators where the particulate concentration or particulate mass emission rate is corrected to 12 percent CO_2, it is recommended that control charts for the field replications and for calibration checks be maintained as given in the quality assurance document of this series for Method 3 (ref. 2).

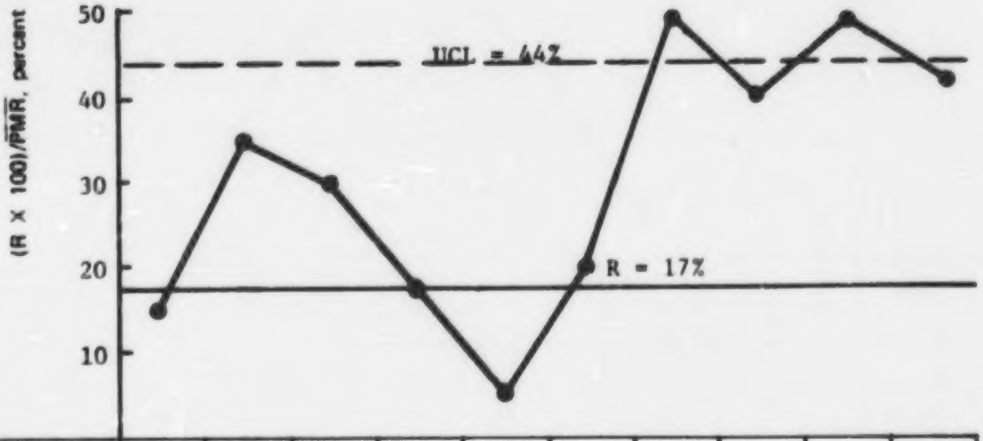
3.3.2.3 Range Chart for Runs. In compliance testing where it is desired to determine the source output at a fixed level of operation, a large range in the three runs (replicates) would suggest process variability and/or measurement variability. Expressing the range, R, as a percent of the average, i.e., the difference in the largest and smallest of the three replicates divided by the average of the three replicates, all multiplied by 100; a control chart with limits as given in Figure 5 can be used initially. Results from collaborative tests of Method 5 show within laboratory CV's of 10, 25, and 31 percent. Therefore, the CV = 10 percent used in constructing the control chart is the least of the three values reported above. These limits should serve as a starting point. When a data point falls out of bounds on this graph, the process data should be checked to see if the process changed between runs and the percent of isokinetic calculations checked to see if one run was significantly different from the others in order to identify the cause of the excessive variability. Note that exceeding the upper control limit does not necessarily invalidate the test data.

3.3.2.4. Mean and Range Charts for Percent of Isokinetic Sampling. Maintaining isokinetic sampling conditions is important in particulate sampling. Control charts displaying the range and mean of the calculated percent isokinetic sampling provide, at a glance, means for evaluating the performance of a team or groups of teams over an extended period of time. If deviations from isokinetic greater than ± 10 percent are not allowed, i.e., the run has to be repeated, then in a rough way 10 percent can be taken as the 3σ value giving a standard deviation of about 3.3 percent. Based on three replicates and the above standard deviation, the range and mean charts for I are given in Figures 6 and 7, respectively.

The R values are plotted sequentially as they are obtained and connected to the previously plotted point with a straight line. Corrective action, such as instruction in proper operating technique, should be taken before the next field test any time one of the following criteria are exceeded:

1. One point falls outside the UCL.
2. Seven consecutive points fall above the $\bar{R}$ line (i.e., the range of I obtained by a given field team is consistently greater than $\bar{R}$ for seven or more field tests). Exceeding the first criteria will usually indicate poor technique or equipment malfunction between sample runs of a particular field test. Exceeding the second criteria indicates a systematic error due to equipment bias or poor technique. (Note that the UCL can be exceeded without violating the $I = 100 \pm 10$ interval for any one run.)

The $\bar{I}$ values, i.e., average percent of isokinetic sampling per three sampling runs, are plotted sequentially as they are obtained from field tests and connected to the previously plotted point with a straight line. Corrective action, such as instruction in proper operating technique

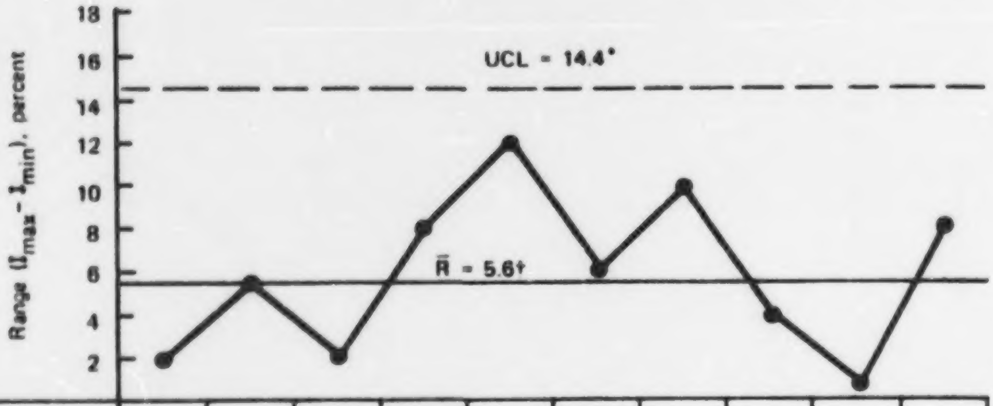


FIELD TEST NO.	1	2	3	4	5	6	7	8	9	10
DATE										
FIELD TEAM										
PROBLEM AND CORRECTIVE ACTION							Process parameters could not be held constant between runs. This is a valid data point.		Percent isokinetic (I) was 130% for run 1. $\overline{PMR}$ < one-half the standard, therefore run was not repeated.	

*UCL = $D_2\sigma$ = 4.358 × 10 = 44

†$\bar{R} = d_2\sigma$ = 1.693 × 10 = 17

Figure 5. Sample control for the range, R, of PMR replicates.

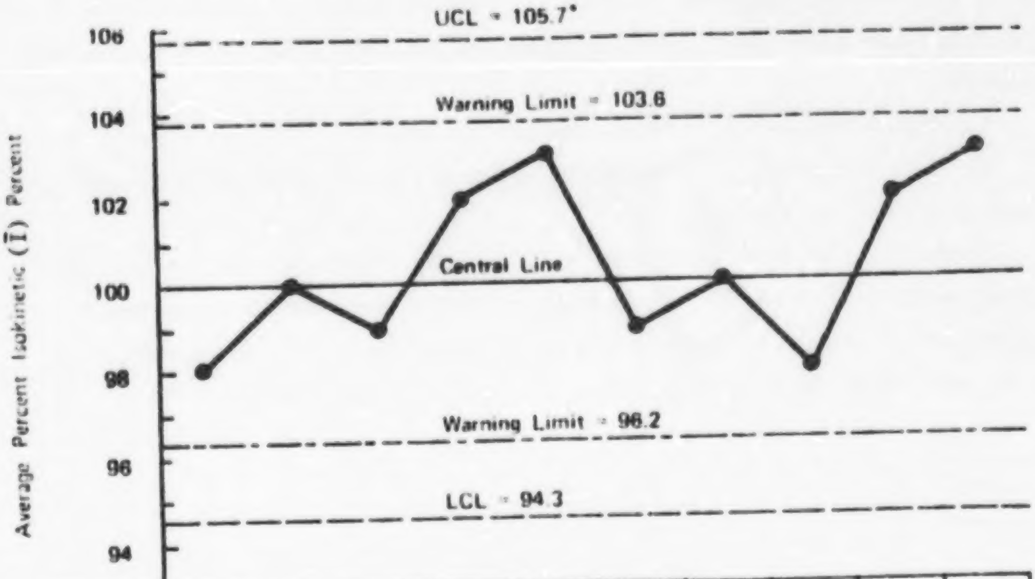


FIELD TEST NO.	1	2	3	4	5	6	7	8	9	10
DATE										
FIELD TEAM										
PROBLEM AND CORRECTION ACTION										

*UCL = $D_2\sigma = 4.358 \times 3.3 = 14.4$

†$\bar{R} = d_2\sigma = 1.693 \times 3.3 = 5.6$

Figure 6. Sample control chart for the range, R, of percent isokinetic, I, sampling for three test runs per field test.



FIELD TEST NO.	1	2	3	4	5	6	7	8	9	10
DATE										
FIELD TEAM										
PROBLEM AND CORRECTIVE ACTION										

*UCL $= \bar{I} + 3\sigma(\bar{I}) = 100 + 3 \times 3.3/\sqrt{3} = 105.7$

Figure 7. Sample control chart for the average percent of isokinetic sampling per field test.

and/or performing equipment calibration checks should be taken before attempting the next field test any time one of the following criteria is exceeded.

1. One point falls outside the UCL or LCL.

2. Two out of three consecutive points fall in the warning zone (between 2σ and 3σ limits).

Exceeding the first criteria will usually indicate poor technique or equipment malfunction. The second criteria when exceeded indicates an assignable source of variability due either to faulty equipment or a consistent error in performing the operation procedures.

SECTION IV MANUAL FOR MANAGER OF GROUPS OF FIELD TEAMS

4.0 GENERAL

The guidelines for managing quality assurance programs for use with Test Method 5 - Determination of Particulate Emissions from Stationary Sources are given in this part of the field document. This information is written for the manager of several teams for measuring source emissions and for the appropriate EPA, State, or Federal Administrators of these programs. It is emphasized that if the analyst carefully adheres to the operational procedures and checks of Section II, then the errors and/or variations in the measured values should be consistent with the performance criteria as suggested. Consequently, the auditing routines given in this section provide a means of determining whether the stack sampling test teams of several organizations, agencies, or companies are following the suggested procedures. The audit function is primarily one of independently obtaining measurements and performing calculations where this can be done. The purpose of these guidelines is to:

1. Present information relative to the test method (a functional analysis) to identify the important operations and factors.

2. Present a methodology for comparing action options for improving the data quality and selecting the preferred action.

3. Present a data quality audit procedure for use in checking adherence to test methods and validating that performance criteria are being satisfied.

4. Present the statistical properties of the auditing procedure in order that the appropriate plan of action may be selected to yield an acceptable level of risk to be associated with the reported results.

These four purposes will be discussed in the order stated in the sections which follow. The first section will contain a functional analysis of the test method with the objectives of identifying the most important factors which affect the quality of the reported data and estimating the expected variation and bias in the measurements resulting from equipment and operator errors.

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Section 4.2 contains several actions for improving the quality of the data; for example, by improved analysis techniques, instrumentation, and/or training programs. Each action is analyzed with respect to its potential improvement in the data quality as measured by its precision. These results are then compared on a cost basis to indicate how to select the preferred action. The cost estimates are used to illustrate the methodology. The manager or supervisor should supply his own cost data and his own actions for consideration. If it is decided not to conduct a data audit, sections 4.1 and 4.2 would still be appropriate as they contain a functional analysis of the reference method and of alternative methods or actions.

There are no absolute standards with which to compare the routinely derived measurements. Furthermore, the taking of completely independent measurements at the same time that the routine data are being collected (e.g., by introducing two sampling probes into the stack and collecting two samples simultaneously) is not considered practical due to the constrained environmental and space conditions under which the data are being collected. Hence, a combination of an on-site systems audit, including visual observation of adherence to operating procedures, and a quantitative performance audit is recommended as a dual means of independently checking on the source emissions data.

The third section contains a description of a data quality audit procedure. The most important variables identified in section 4.1 are considered in the audit. The procedure involves the random sampling of n stacks from a lot size of N = 20 stacks (or from the stacks to be tested during a three-month period, if less than 20) for which one firm is conducting the source emissions tests. For each of the stacks selected, independent measurements will be made of the indicated variables. These measurements will be used in conjunction with the routinely collected data to estimate the quality of the data being collected by the field teams.

The data quality audit procedure is an independent check of data collection and analysis techniques with respect to the important variables. It provides a means of assessing data collected by several teams and/or firms with the potential of identifying biases/excessive variation in the data collection procedures. A quality audit should not only provide an independent quality check, but also identify the weak points in the measurement process. Thus, the auditor, an individual chosen for his background knowledge of the measurement process, will be able to guide field teams in using improved techniques. In addition, the auditor is in a position to identify procedures employed by some field teams which are improvements over the current suggested ones, either in terms of data quality and/or time and cost of performance. The auditor's role will thus be one of aiding the quality control function for all field teams for which he is responsible, utilizing the cross-fertilization of good measurement techniques to improve the quality of the collected and reported data.

The statistical sampling and test procedure recommended is sampling by variables. This procedure is described in section 4.4. It makes maximum use of the data collected, and it is particularly adaptable to the small lot size and, consequently, the small sample size applications. The same sampling plans can be employed in the quality checks performed by a team or firm in its own operations. The objectives of the sampling and test procedure are to characterize data quality for the user and to identify potential sources of trouble in the data collection process for the purpose of correcting the deficiencies in data quality.

Section 4.4.4 describes how the level of auditing, i.e., the sample size n, may be determined on the basis of relative cost data and prior information about the data quality. This methodology is described in further detail in the Final Report on the Contract. The cost data and prior information concerning data quality are supplied to illustrate the procedure and these data must be supplied by the manager of groups of field teams depending upon the conditions particular to his responsibility.

Figure 8 provides an overall summary of the several aspects of the data quality assurance program as described in these documents. The flow diagram is subdivided into four areas by solid boundary lines. These areas correspond to specific sections or subsections of the document as indicated in the upper right hand corner of each area. The details are considered in these respective sections of the document and will not be described here.

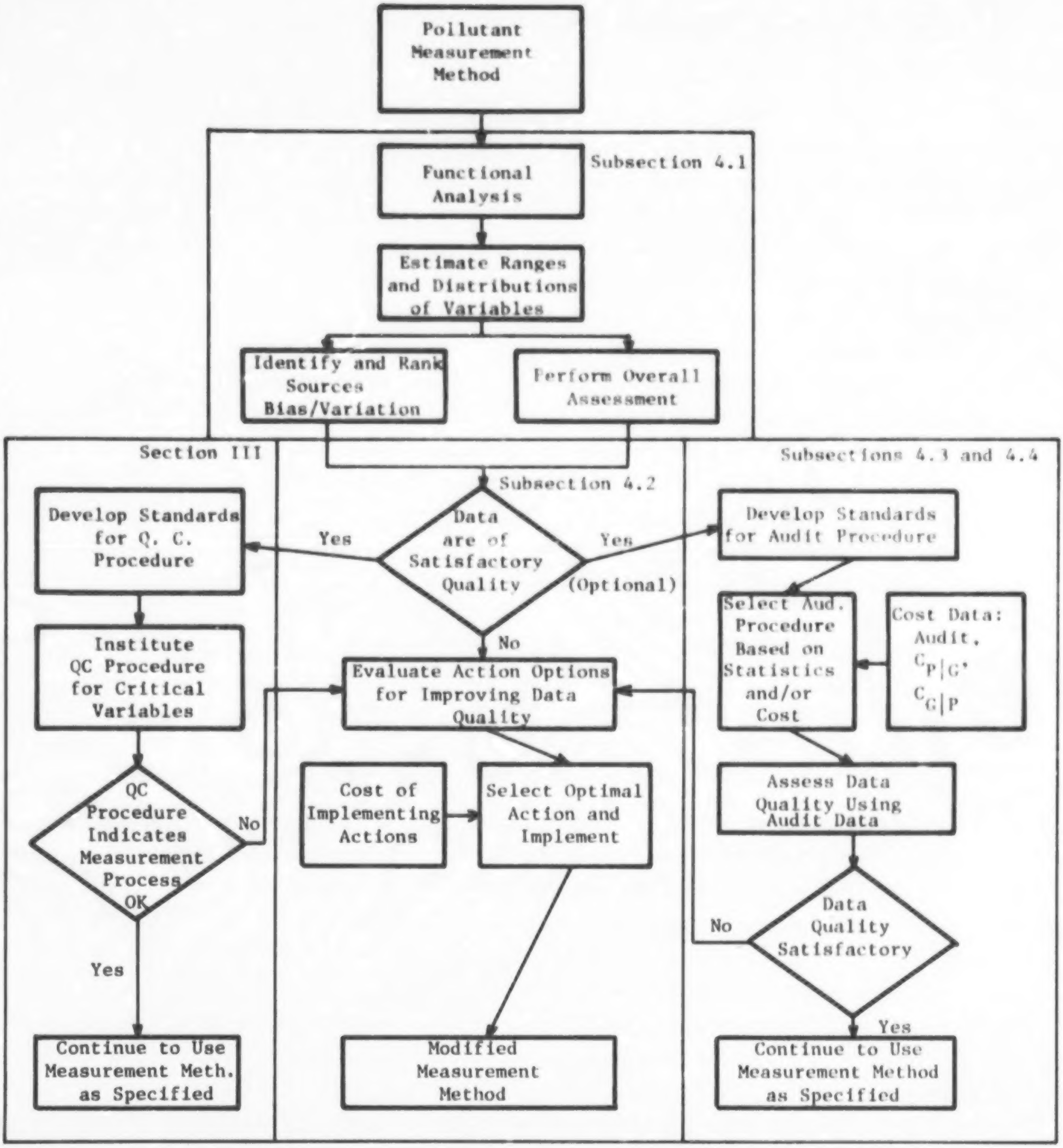

Pollutant Measurement Method
Functional Analysis
Subsection 4.1
Estimate Ranges and Distributions of Variables
Identify and Rank Sources Bias/Variation
Perform Overall Assessment
Section III
Subsection 4.2
Subsections 4.3 and 4.4
Develop Standards for Q. C. Procedure
Yes
Data are of Satisfactory Quality
Yes
(Optional)
Develop Standards for Audit Procedure
No
Institute QC Procedure for Critical Variables
Evaluate Action Options for Improving Data Quality
Select Aud. Procedure Based on Statistics and/or Cost
Cost Data: Audit, $C_{P|G}$, $C_{G|P}$
QC Procedure Indicates Measurement Process OK
No
Cost of Implementing Actions
Select Optimal Action and Implement
Assess Data Quality Using Audit Data
No
Data Quality Satisfactory
Yes
Yes
Continue to Use Measurement Meth. as Specified
Modified Measurement Method
Continue to Use Measurement Method as Specified

Figure 8. Summary of data quality assurance program.

4.1 FUNCTIONAL ANALYSIS OF TEST METHOD

Test Method 5 - Determination of Particulate Emissions from Stationary Sources--as described in Appendix A of this document is subjected to a functional analysis in this section. This measurement method is used to determine the concentration of particulate matter in the stack gas. It is also used to simultaneously determine the moisture content of the stack gas and to perform a velocity traverse from which the volumetric flow rate is calculated. These results combined with the stack gas composition as measured by Method 3 yield a particulate mass emission rate for the source being tested.

Method 5 as promulgated in the Federal Register, Vol. 36, No. 247 - Thursday, December 23, 1971 has been subjected to collaborative tests on a Portland cement plant (ref. 16), a fossil fuel-fired steam generator (ref. 17), and a municipal incinerator (ref. 18). These tests showed within laboratory precisions (expressed as coefficient of variation) ranging from 10 to 30 percent and between laboratory precisions of 20 to 40 percent. Several recommendations were made for improving the precision of the method based on the collaborative test results (refs. 16, 17 and 18).

The method (i.e., Method 5) has been revised by EPA incorporating the above mentioned recommendations as well as other changes that should improve the precision of the method. The revised method is reproduced as Appendix A of this document. The functional analysis presented in the following subsections is based on what can hopefully be achieved using the revised method. A within laboratory coefficient of variation of 10 percent may be achievable by highly motivated, well-trained, stack sampling teams.

The changes and/or additions made by EPA to the method that should result in significant improvement in data quality include:

1. Minimum free spaces between the pitot tube, sampling nozzle, and temperature sensor are specified (see Figure 5-1 in Appendix A).

2. Calibration of the type-S pitot tube is required prior to initial use and at any sign of damage, plus the calibration standard, facility and procedures are fully specified.

3. Greater detail on sample recovery is provided.

4. Sampling train leak checks are required before and after a run and are recommended between port changes.

The purposes of the functional analysis as performed here are to:

1. To identify operations, variables and factors that can influence the quality of the measurement data.
2. To illustrate how elements of uncertainty are propagated through the measurement process to the resultant particulate measurement.
3. To estimate the mean values and ranges of the various error sources existing under normal operating conditions and to determine the combined probable uncertainty to be associated with the reported measurements.

The results of the functional analysis provide information for the following:

1. Setting acceptable limits on data quality, i.e., precision and accuracy.
2. Applying quality control procedures to those operations and variables that would otherwise result in excess variability in the measurement data.
3. Applying appropriate quality assurance practices (e.g., audits) at critical points in the measurement process with acceptable limits specified and actions to be taken when limits are exceeded.

The functional analysis is discussed in two stages. First the variables and operations involved in the measurement process are identified and modeled, i.e., their mean values and variances are estimated. These estimates are made using published data if available or engineering judgment if no quantitative data are available. Second, a variance analysis (sometimes referred to in the literature as a sensitivity analysis or error analysis) is performed to determine the individual and combined influence of the variables and operations to the particulate mass emission rate measurement.

The variance analysis as presented in this section provides a mathematical means of identifying important variables in the measurement process. The estimates of the mean values and variances of the variables are made as realistic as possible, however, the major purpose of this section is to illustrate a method. Each laboratory should work through the analysis using experimental data or estimates based on conditions within that laboratory. The mathematical basis for the procedures are described in the Final Report on Contract 68-02-0598 (ref. 4).

The particulate mass emission rate (PMR) is calculated from the relationship:

$$PMR = 10^{-3} \text{ g/mg } (m_n/V_{m(std)}) \, (V_s \cdot A_s) \qquad (1)$$

where

PMR = Rate of particulate emissions, g/hr.
m_n = Total weight of particulate collected, mg.
$V_{m(std)}$ = Volume of gas sampled corrected to standard conditions, m^3.
V_s = Stack gas velocity, m/s.
A_s = Crossectional area of stack, m^2.

Variability in the measured PMR will have components of variability from the individual determinations of m_n, $V_{m(std)}$, V_s and A_s. Variability of each of the four terms is discussed separately below following a short definition of terms.

Many different measures of variability are conceivable depending upon the circumstances under which the measurements are performed. Three measures of variability are defined here. They are replicability, repeatability and reproducibility defined as follows (ref. 22).

1. Replicability. The precision measuring the variability among replicates. Replicates are repeated but independent determinations of the same sample, by the same analyst, at essentially the same time and same conditions. This could apply to the three runs comprising a field test provided the process variables remained constant.

2. Repeatability is a measure of the variability in determinations made on the same sample (i.e., the same source with source parameters held as constant as possible) by the same field team using the same equipment over a short period of time. The repeatability standard deviation and the coefficient of variation are symbolized by σ_r and CV_r respectively. This

measure of precision is referred to as the within laboratory standard deviation in the collaborative test reports.

3. Reproducibility is the variability associated with measurements made of the same sample (i.e., same source with source parameters held as constant as possible) by field teams from different laboratories. This measure of precision is referred to as between laboratory precision in the collaborative test reports.

The above definitions are based on a statistical model where each determination of the rate of emissions is the sum of three components as follows:

$$PMR = \overline{\overline{PMR}} + b + e \qquad (2)$$

where

PMR = the measured rate of particulate emissions, g/hr.

$\overline{\overline{PMR}}$ = the general or true average, rate of particulate emissions, g/hr.

b = an error term representing the differences between field teams equipment, sources, etc., g/hr.

e = a random error occurring in each measurement, g/hr.

In general, b can be considered as the sum:

$$b = b_r + b_s \qquad (3)$$

where

b_r = random component

b_s = systematic component

The term b is considered to be constant during any series of measurements performed under replicability conditions, e.g., the three runs making up a field test. It behaves as a random variate in a series of measurements during which personnel, equipment or conditions change e.g., between field tests or laboratories. Its variance is denoted by

$$\text{var } b = \sigma_b^2 \qquad (4)$$

where σ_b^2 is the bias component of variability due to different field teams, equipment, etc.

The term e represents a random error occurring in each measurement. Its variance is denoted by

$$\text{var } e = \sigma^2 \tag{5}$$

where σ^2 is called the replicability variance.

The repeatability standard deviation, σ_r, is related to the bias variance and the replicability variance and the number of replicates r by

$$\sigma_r = \sqrt{\sigma^2/r + \sigma_b^2} \tag{6}$$

Repeatability of particulate mass emission rate determinations will be discussed in the following subsections.

4.1.1 Mass of Particulate Collected, m_n

The total measured particulate mass, m_n, is given by

$$m_n = m_t + \varepsilon, \tag{7}$$

where m_t represents the true particulate mass in the gas volume at stack conditions equivalent to the volume $V_{m(std)}$ as measured by the dry gas meter and corrected to standard conditions. The difference between the true and measured values is the error, ε.

Determination of particulate mass is subject to error from

1. Loss or gain of sample during sample recovery (SR),
2. Weighing of collected sample (W),
3. Conversion of acid gases to particulates by alkaline glass fiber filters (pH),
4. Formation of particulates due to non-uniform or inadequate heating of the probe and/or sample box (C), and
5. Deviation from isokinetic sampling (I).

These five components of variability are independent (at least there are no obvious correlations) and with each component's effect an m_n given in mg the variance of m_n becomes:

$$\sigma^2\{m_n\} = \sigma^2_{SR}\{m_n\} + \sigma^2_W\{m_n\} + \sigma^2_{pH}\{m_n\} + \sigma^2_c\{m_n\} + \sigma^2_I\{m_n\}. \tag{8}$$

Magnitudes of each of the five variance components are discussed in the following subsections. Using the variances, as estimated in the following subsections, in the above relationship gives a total variance for m_n of

$$\sigma^2\{m_n\} = 0.0016 \times m_t^2 + 0.0016 \times m_t^2 + 9\ \mu g^2 + 4\ \mu g^2 + 0.0034 \times m_t^2.$$

By rearranging and combining terms the variance becomes

$$\sigma^2\{m_n\} = 0.0066\ m_t^2 + 13\ mg^2. \tag{9}$$

Values of $\sigma\{m_n\}$ and $CV\{m_n\}$ for different values of true particulate mass, m_t, are given in a table in subsection 4.1.1.6.

4.1.1.1 Sample Recovery (SR). Error due to a loss or gain of sample during sample recovery is mostly a function technique and of the adequacy of the work area used for sample recovery. Sample recovery includes any excess sample due to scraping the probe against the stack wall or sampling port as well as transferring the filter and collected particulate from the filter holder to a sample container and removing particulate matter from the nozzle, probe, filter holder, and connecting glassware. No quantitative data are available for estimating the magnitude of this error. However, it was considered to be a major source of error by those conducting the collaborative tests (refs. 16, 17, and 18). For this analysis an error proportional to the true mass, m_t, is assumed. A relative error (coefficient of variation) of 4 percent given a stand deviation of $\sigma_{SR}\{m_n\} = 0.04 \times m_t$ mg and a variance of $\sigma^2_{SR}\{m_n\} = 0.0016 \times m_t^2\ mg^2$. It is further assumed that over a large number of field tests there will be as many positive as negative errors i.e., this error can be modeled as a normally distributed variate having a zero mean and a variance of $\sigma^2_{SR}\{m_n\}$. This is symbolized as $N(\mu, \sigma^2) = N(0, 0.0016\ m_t^2)$.

4.1.1.2 Weighing Errors (W). Sample weighing errors are a combination of four weighings. The weight of particulate matter collected m_n is

$$m_n = (W_2 - W_1) + (W_4 - W_3) \tag{10}$$

where

m_n = The collected mass of particulate matter, mg
W_1 = The tare weight of the filter, mg
W_2 = The weight of the exposed filter, mg
W_3 = The tare weight of a beaker or drying dish, mg, and
W_4 = The gross weight of the beaker and the particulate material collected in the probe, mg.

If the recommended procedures are followed for checking the balance measurements against measurement standards before each weighing, the associated weighing errors for each of the weighings, W_1, W_2, W_3, and W_4 would most likely result from improper conditioning and handling rather than from the balance. Under this assumption the weighings are uncorrelated and the variances can be combined as follows.

$$\sigma_w^2 \{m_n\} = \sigma^2\{W_1\} + \sigma^2\{W_2\} + \sigma^2\{W_3\} + \sigma^2\{W_4\} \tag{11}$$

Weighings W_1 and W_3 are tare weighings and should have greater precision than weighings W_4 and W_2 which involved the particulates recovered from the probe assembly and the filter, respectively. These latter weighings are sensitive to the type of particulates i.e., if they are hygroscopic (ref 17) or of high organic content. Rather than estimate the individual weighing errors the total standard deviation of m_n attributable to weighing operations is estimated to be 4 percent of the true particulate mass, i.e., $\sigma_w\{m_n\} = 0.04 \times m_t$ mg or a variance of $\sigma_w^2\{m_n\} = 0.0016\ m_t^2$.

4.1.1.3 Filter Surface Alkalinity (pH). The surface of glass fiber filters is generally alkaline with pH's ranging from about 7 to 11. Acid gases in the sample air can be oxidized and deposited as particulate matter on these filters. The quantity of pseudo-particulates formed is probably a function of the filter pH, concentration of the acid gases in the sample gas, residence time, and duration of the sampling period. Tests dealing with measuring suspended particulates in ambient air showed a much higher particulate catch when using filters with a pH of 11 as compared to filters with a pH of 6.5 (refs. 23 and 24). One series of nine tests involving samplers operated side by side, one with a pH-11 filter and the other with a pH-6.5 filter, showed an average particulate catch 18 percent greater for the alkaline filters (ref. 23). A glass fiber filter used as a backup to a cloth primary filter in a sampling train showed a weight gain due to soluble sulfates as large as the total particulate catch of the primary filter (ref. 25).

The above data on alkaline fi-ters are not directly applicable to this application, thus an error limit would be strictly an estimate. For this analysis a range of from 0 to 9 mg is assuemd as the potential gain in particulate matter due to conversion of acid gases on the filter surface. This error acts as a positive bias and is modeled as the absolute value of a normal distribution with a zero mean and a variance of 9 mg^2 or a standard deviation of $\sigma_{pH}(m_n) = 3$ mg, i.e., $|N(0, 9 \text{ gm})|$. Such a distribution (taking absolute values) has a mean value of approximately + 2.4 mg and cannot have a negative value.

4.1.1.4 Condensation (C). Hemeon and Blank (ref. 25) discuss the possibility of pseudo-particulate matter being formed in the probe prior to the filter by oxidation of SO_2 in the sample gas. This could occur if the probe is not uniformly heated and condensation occurs, followed by heating and revaporization. Also, many compounds pass from the gaseous to the solid phase between 93°C to 148°C (200 to 300°F) (ref. 26); thus, sampling trains performing at different operating temperatures could generate biased data. Insufficient data are available to model this source of error. Therefore, just to include it as a significant error, it is treated as a normally distributed random variable with a 4 mg mean and a standard deviation of 2 mg, i.e., $N(4 \text{ mg}, 4 \text{ mg}^2)$. Under these conditions, it acts generally as a positive bias but could go negative simulating a probe operating above 120°C (248°F), for example.

4.1.1.5 Nonisokinetic Sampling (I). Deviation from isokinetic sampling conditions may result in particulate measurement error. The magnitude of error depends on the degree of departure from isokinetic conditions, and on the particle size distribution in the sample gas. Departure from isokinetic sampling can occur due to failure to adjust the nozzle velocity as the stack gas velocity varies. This type of departure will be detected when the percent of isokinetic sampling is calculated. However, errors in the pitot tube coefficient and/or in the nozzle diameter can cause deviations from isokinetic conditions which are not detectable from any checks that can be performed while in the field.

The degree of deviation from isokinetic sampling is not a direct indication of the error in the final result. The error resulting from nonisokinetic sampling is a function of the particle size distribution in the stack. For gases and small particles (diameters ≤ 5 μm) isokinetic sampling is not necessary (ref. 27). Sample gases with particle size distributions extending in the 25 μm and above sizes are sensitive to anisokinetic sampling. Each particle size distribution has to be evaluated. For this analysis, using a particle size distribution of 80 to 100 μm particles (ref. 19) a variation of ± 5 percent from isokinetic sampling shows a relative variation of approximately ± 10 percent in the collected mass or a ratio of 2 to 1. For this analysis a 1 percent deviation from isokinetic conditions is taken to be a 2 percent error in the measured particulate concentration, i.e.,

$$\sigma_I\{m_n\} = 2 \times CV\{I\} \times m_t. \tag{12}$$

Percent of isokinetic sampling is derived from the ratio of the gas velocity in the nozzle to the stack gas velocity in the stack by

$$I = V_n/V_s. \tag{13}$$

The component of variability in m_n due to nonisokinetic sampling is taken as

$$\sigma_I\{m_n\} = 2 \times CV\{V_n/V_s\} \times m_t. \tag{14}$$

The coefficient of variation $CV\{V_n/V_s\}$ is derived in the following manner.

The estimated coefficient of variation of V_n based on

$$V_n = \frac{4\ K_m}{\pi D_n^2} \left[\frac{1 - B_{wm}}{1 - B_{ws}}\right]\left[\frac{T_m\ \Delta H}{P_m\ M_m}\right]^{1/2} \tag{15}$$

is given by

$$\begin{aligned} CV^2\{V_n\} \simeq\ & CV^2\{K_m\} + 4CV^2\{D_n\} + CV^2\{1 - B_{wm}\} \\ & + CV^2\{1 - B_{ws}\} + 0.25(CV^2\{T_m\} + CV^2\{\Delta H\} \\ & + CV^2\{P_m\} + CV^2\{M_m\}). \end{aligned} \tag{16}$$

where

V_n = Sample gas velocity in the nozzle.
D_n = Average nozzle diameter.
B_{wm} = Water vapor in sample gas at the dry gas meter (taken as zero for this analysis).
B_{ws} = Water vapor in the sample gas by proportion.
T_m = Absolute temperature at the dry gas meter.
P_m = Ambient pressure at the meter.
M_m = Molecular weight of the sample gas at the meter.
ΔH = Pressure drop across the sampling train orifice.
K_m = A constant whose relative error depends on the errors in the individual measured values in the equation.

Table 2 gives the assumed mean values and the estimated coefficients of variation of the individual parameters in the above equation.

Substituting the estimated CV's from Table 2 into the above equation 16 yields a coefficient of variation for V_n of

$$CV\{V_n\} = 2.04\%.$$

Table 2. Means and variabilities of parameters affecting V_n

Parameter	Mean Value Metric	(English)	Estimated CV
K_m	1.165×10^{-3}	(1.84)	$CV\{K_m\} = 1.0\%$
D_n	.635 cm	(0.25 in)	$CV\{D_n\} = 0.5\%$
$1 - B_{wm}$	0.0	(0.0)	0.0
$1 - B_{ws}$	0.90	(0.90)	$CV\{1 - B_{wo}\} = 1.2\%$
T_m	294°K	(530°R)	$CV\{T_m\} = 0.5\%$
ΔH	102 mm H_2O	(4.0 in H_2O)	$CV\{\Delta H\} = 1.0\%$
P_m	762 mm Hg	(30.0 in Hg)	$CV\{P_m\} = 0.3\%$
M_m	30 g/g-mole	(30 lb/lb-mole)	$CV\{M_m\} = 1.0\%$
V_s	15.24 m/sec	(50 ft/sec)	$CV\{V_s\} = 2.07\%$
V_n	15.24 m/sec	(50 ft/sec)	$CV\{V_n\} = 2.04\%$

A functional analysis of Method 2--Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S pitot tube)--was made and reported in reference 2. The results of that analysis is used here without repeating the analysis. A coefficient of variation of

$$CV\{V_s\} = 2.07\%$$

was derived for the stack gas velocity determination.

Using $CV\{V_n\}$ and $CV\{V_s\}$ the coefficient of variation for the ratio V_n/V_s becomes

$$CV\{V_n/V_s\} = \sqrt{CV^2\{V_n\} + CV^2\{V_s\}} \qquad (17)$$

or

$$CV\{V_n/V_s\} = 2.91\%$$

The average value of $V_n/V_s = \bar{I} = 100$, therefore

$$CV\{I\} = 2.91\%.$$

The component of variability of m_n due to nonisokinetic sampling as given by equation 12 becomes

$$\sigma^2\{m_n\} = 0.0034 \times m_t^2 ,$$

yielding a standard deviation of

$$\sigma_I\{m_n\} = 0.0582 \times m_t . \qquad (18)$$

4.1.1.6 Summary of Errors in Measured Particulate Mass, m_n. Using the variance components derived in subsections 4.1.1.1 through 4.1.1.5, the variance of m_n is

$$\sigma^2\{m_n\} = 0.0066\ m_t^2 + 13\ mg^2. \qquad (19)$$

The variance, standard deviation, and coefficient of variation for different values of m_t are given in the following table

m_t	$\sigma^2\{m_n\}$	$\sigma\{m_n\}$	$CV\{m_n\}$
100 mg	79 mg^2	8.9 mg	8.9%
200 mg	277 mg^2	16.6 mg	8.3%
400 mg	1069 mg^2	32.7 mg	8.2%
600 mg	2389 mg^2	48.9 mg	8.1%

4.1.2 Volume of Gas Measured by the Dry Gas Meter and Corrected to Standard Conditions, $V_{m(std)}$.

The sample volume measured by the dry gas meter and corrected to standard conditions is given by (equation 5-1 is Appendix A).

$$V_{m(std)} = K\ V_m \left[\frac{P_{bar} + \Delta H/13.6}{T_m} \right] \qquad (20)$$

(See definitions of terms in Section 6.1 of Appendix A.)

Variability in V_m is a combination of variability of the dry gas meter, inaccuracy of the calibration standard (i.e., wet test meter), and sampling train leaks, including those leaks undetected because of pump valve float. Assuming coefficients of variation of 1 percent, 0.4 percent, and 0.5 percent for the dry gas meter, calibration standard, and sampling train leaks, respectively, results in a $CV\{V_m\} = 1.2$ percent.

$CV\{P_{bar}\} = 0.3$ percent and $CV\{T_m\} = 0.5$ percent are assumed from previous documents of this series (ref. 2).

Variability in ΔH is believed to be primarily due to reading error (inclined manometer) and calibration error in determining $\Delta H@$ ($\Delta H@$ is the pressure drop across the orifice meter resulting in a 354 cm^3/sec (0.75 ft^3/min) flow rate at standard temperature and pressure). The pressure drop across the orifice, i.e., ΔH, relatively constant with no fluctuations and hence, easily read on an inclined manometer. Also, random reading error is averaged out because ΔH as used in the equation is an average of at least 12 readings. Therefore, the variability of the average pressure drop should be adequately characterized by a coefficient of variation of 1 percent, i.e., $CV\{\Delta H\} = 1$ percent. These assumptions are summarized in table 3. The estimated variance of $V_{m(std)}$, as given by equation 20, is

$$\sigma^2\{V_{m(std)}\} \simeq 1.004\ \sigma^2\{V_m\} + 4.02\ \sigma^2\{P_{bar}\} + 0.0129\ \sigma^2\{T_m\} + 0.0217\ \sigma^2\{\Delta H\}. \quad (21)$$

Using the estimated means and standard deviations of table 4 (in English units) the estimated variance of $V_{m(std)}$ is $\sigma^2\{V_{m(std)}\} \simeq 0.644$ and the standard deviation $\sigma\{V_{m(std)}\} \simeq 0.8\ ft^3$. The estimated mean is

$$\bar{V}_{m(std)} = 1.7\ m^3\ (60\ ft^3)$$

resulting in a coefficient of variation of 1.3 percent ($= 0.8 \times 100/60$). The most important variable in the determination of $V_{m(std)}$ under the stated assumptions is clearly V_m.

Table 3. Means and variabilities of parameters in determining the sample gas volume

Parameter	Mean value	Coefficient of variation	Standard deviation
V_m	$\bar{V}_m = 1.70\ m^3$ $(60\ ft^3)$	$CV\{V_m\} = 1.2\%$	$\sigma\{V_m\} = 0.02\ m^3$ $(0.72\ ft^3)$
P_{bar}	$\bar{P}_{bar} = 760$ mm Hg (29.92 in. Hg)	$CV\{P_{bar}\} = 0.3\%$	$\sigma\{P_{bar}\} = 2.3$ mm Hg (0.09 in. Hg)
T_m	$\bar{T}_m = 294°K$ (530°R)	$CV\{T_m\} = 0.5\%$	$\sigma\{T_m\} = 1.5°K$ (2.65 °R)
ΔH	$\Delta H = 100.6$ mm H_2O (4 in. H_2O)	$CV\{\Delta H\} = 1.0\%$	$\sigma\{\Delta H\} = 1.0$ mm H_2O (0.04 in. H_2O)

4.1.3 Volumetric Flow Rate, Q_s.

A functional analysis of the method used to measure volumetric flow rate was made in the quality assurance guidelines document for Method 2 (ref. 1). Volumetric flow rate is given by the product of the stack gas velocity, V_s, and the cross sectional area of the stack, A_s. The results of that analysis will be used here without repeating the analysis itself. A coefficient of variation of 2.33 percent was derived for the volumetric flow rate.

That is,

$$CV\{Q_s\} = CV\{V_s \cdot A_s\} = 2.33\%.$$

4.1.4 Precision of Particulate Concentration Determinations, C_s.

The concentration of particulate matter in the stack gas, g/m^3, on a dry basis corrected to standard conditions, is given by

$$C_s = (10^{-3}\ g/mg)\ (m_n/V_{m(std)}).$$

Using previously derived results, the estimate precision of C_s is given by

$$\sigma^2\{C_s\} = \sigma^2\{m_n\} + \sigma^2\{V_{m(std)}\} \qquad (22)$$

where $\sigma^2\{m_n\}$ and $\sigma^2\{V_{m(std)}\}$ are given by equations (19) and (21), respectively. Substituting these results into (22) and using values of 100, 200, and 300 mg for m_t yields:

m_t	$\sigma\{C_s\}$	$CV\{C_s\}$
100 mg.	9.0 mg.	9.0%
200 mg.	16.8 mg.	8.4%
400 mg.	33.2 mg.	8.3%

Variability in m_n is the dominating source of variability in the determination of C_s.

4.1.5 Precision of Particulate Mass Emission Rate, PMR.

The particulate mass emission rate is calculated by

$$PMR\ (g/hr) = Q_s\ (m^3/hr) \times C_s\ (g/m^3)$$

and

$$CV^2\ \{PMR\} = CV^2\ \{Q_s\} + CV^2\ \{C_s\}. \qquad (23)$$

$CV\{Q_s\}$ was derived in the Quality Assurance Document for Method 2 (ref. 1) as 2.33 percent. Inserting this value and the estimated values of $CV\{C_s\}$ from the preceding subsection into equation (23), results in the following CVs of the PMR measurements for $\bar{V}_{m(std)} = 1.7\ m^3$.

m_t	CV {PMR}
100 mg.	9.3%
200 mg.	8.7%
400 mg.	8.6%

The concentration factor, C_s, is the more important of the two variables in equation (23), accounting for about 80 percent of the total variability in PMR.

4.1.6 Summary of Functional Analysis.

In summary, the most important variables in the determination of PMR are identified in the diagram of figure 9. The coefficient of variation for each of the important variables is given and a flow diagram indicating how each variable influences the pollutant mass emission rate is given in the figure. Table 4 summarizes the variance analysis calculations. Results for three levels of collected particulate mass are given for concentration and for particulate mass emission rate.

4.1.7 Bias Analysis

Two bias terms were identified in the functional analysis of section 4.1.1. They are the error in the collected mass m_n due to (1) filter surface alkalinity, and (2) condensation in the probe prior to the filter by oxidation of SO_2 in the sample gas. Combining the estimated biases of these two effects from sections 4.1.1.3 and 4.1.1.4, respectively, yields the following overall bias in m_n values.

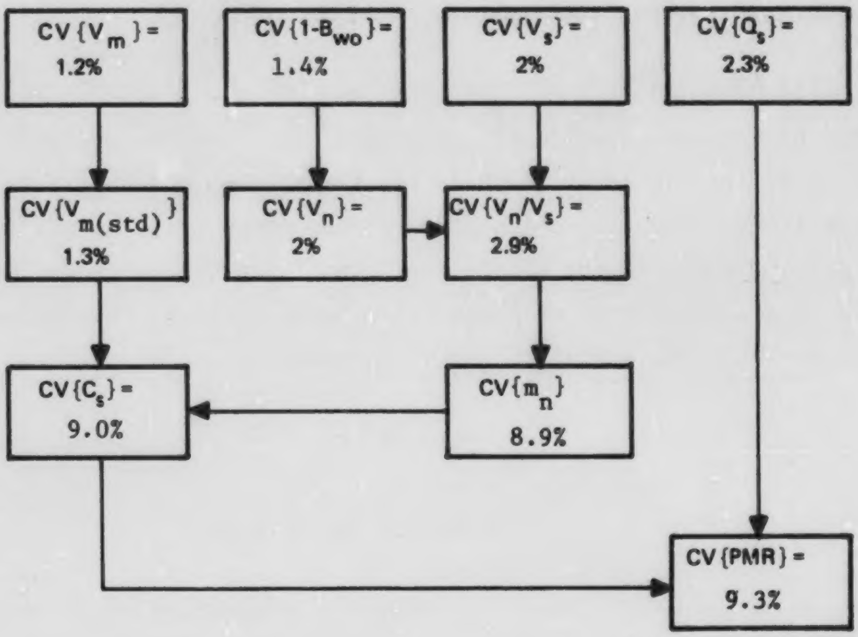
$$\hat{\tau}\{m_n\} = \hat{\tau}\{pH\} + \hat{\tau}\{C\} \tag{24}$$

$$= 2.4 \text{ mg} + 4 \text{ mg} = 6.4 \text{ mg}.$$

For $m_t = 100$ mg, the bias represents an overall potential relative bias of +6.4 percent. However, for $m_t = 400$ mg, the relative bias is only +1.8 percent (= 100 × 6.4 mg/400 mg). Thus, the bias could be significant for the small values of true particulate mass, m_t.

4.2 ACTION OPTIONS

Suppose it has been determined as a result of the functional analysis and/or the reported data from the checking and auditing schemes, that the data quality is not consistent with suggested standards or with the user requirements. Poor data quality may result from (1) a lack of adherence to the control procedures given in section II--Operations Manual, or (2) the need for an improved method or instrumentation for taking the measurements. It is assumed in this section that (2) applies; that is, the data quality needs to be improved beyond that attainable by


CV{V$_m$} = 1.2%
CV{1-B$_{wo}$} = 1.4%
CV{V$_s$} = 2%
CV{Q$_s$} = 2.3%
CV{V$_{m(std)}$} 1.3%
CV{V$_n$} = 2%
CV{V$_n$/V$_s$} = 2.9%
CV{C$_s$} = 9.0%
CV{m$_n$} 8.9%
CV{PMR} = 9.3%

*CV{C_s} determined for m_t = 100 mg.

Figure 9. Relationship of the most important intermediate measurements to the determination of PMR.

Table 4. Summary of results of variance analysis

Variable	Statistic	m_t		
		100 mg	200 mg	400 mg
$V_{m(std)}$ (m^3)	Mean	←	60.6 *	→
	σ Eq. (21)	←	0.80	→
	CV(%)	←	1.33%	→
V_n (m^3)	Mean	←	63.9 *	→
	σ	←	1.30	→
	CV(%) Eq. (16)	←	2.04%	→
V_n/V_s	Mean	←	1 *	→
	σ	←	0.029	→
	CV(%) Eq. (17)	←	2.91%	→
m_n (mg)	Mean	100	200	400
	σ Eq. (8)	8.9	16.6	32.7
	CV(%)	8.9%	8.3%	8.2%
Nonincinerator case				
C_s	Mean	5.83×10^{-8} g/m^3	11.7×10^{-8} g/m^3	23.39×10^{-8} g/m^3
	σ Eq. (22)	0.51×10^{-8} g/m^3	0.98×10^{-8} g/m^3	1.94×10^{-8} g/m^3
	CV(%)	9.0%	8.4%	8.3%
PMR_c	Mean			
	σ			
	CV(%) Eq. (23)	9.3%	8.7%	8.6%

---

[*] Results do not depend on m_t.

following the operational procedures given for the reference method.

The selection of possible actions for improving the data quality can best be made by those familiar with the measurement process and by the judicious use of the functional analysis as described in section 4.1. For each action, the variance analysis can be performed to estimate the variance, standard deviation, and coefficient of variation of the pertinent measurement(s). In some cases it is difficult to estimate the reduction in specific variances, which are required to estimate the precisions of the pertinent measurements. In such cases, an experimental study should be made of the more promising actions based on preliminary estimates of precision/bias and costs of implementation of each action. This preliminary analysis would follow the methods suggested herein.

A0: Reference Method
A1: Compute PMR by sample concentration and ratio of area methods and average (cost of \$200/20 stacks)(ref. 23).
A2: Crew training (cost of \$1000/20 stacks)
A3: Use modified ORSAT (for incinerator)(cost of \$200/20 stacks)
A4: Use programmable minicalculator in lieu of nomographs (cost of \$350/20 stacks).

The costs given for each action are additional costs above that of the reference method. The assumptions made concerning the reduction in the variances (or improved precisions), are given in the following paragraphs for each action.

1. Compute PMR by Sample Concentration and by Ratio of Area Methods and Average, A1. It is assumed that the error associated with anisokinetic sampling is reduced to one-half by this action as compared to the value calculated by the reference method i.e., calculating by sample concentration only (ref. 23). This assumption results in a reduction in the relative standard deviation (CV) of $\overline{\text{PMR}}$ from 9.3 percent to 5.0 percent for $m_t = 100$ mg.

A nominal cost of \$200 per 20 stacks is used as the cost of implementing this action. Calculations for the particulate mass emission rate by the method of sample concentration, PMR_c, and for the percent of isokinetic sampling, $I = V_n/V_s$, are required by the reference method. The product of PMR_c and I yields the particulate mass emission rate by the ratio of areas method, PMR_a. Hence, very little additional effort is required.

2. Crew Training, A2. It is assumed that the coefficient of variation of PMR is reduced by 25 percent or,

$$CV\{PMR|A2\} = 0.75\ CV\{PMR|A0\}.$$

The notation CV{PMR|A2} denotes the estimated CV of PMR given that action A2 is implemented; the vertical line being read given that, and action following the vertical line denotes the action implemented. This results in a straightforward computation of the CVs,

m_t	CV {PMR|A0}	CV {PMR|A2}
100 mg.	9.3%	7.0%

In estimating a cost for implementing this action it was assumed that one crew member sent to a source sampling school such as that conducted by the EPA for 1 week out of a year would constitute special crew training. The cost of the school plus subsistence for a week was estimated to be about 1,000 dollars. This is in excess of salary and benefits. The cost was prorated over 20 source tests which was taken as a reasonable number of tests per team per year.

3. Use Modified ORSAT (for incinerator sources), A3. It is assumed that the standard deviation of the measurement of $\%CO_2$ is reduced from 0.4 percent to 0.2 percent (absolute). For small values of $\%\overline{CO}_{2_t}$ the true mean percent CO_2, the effect of the error in the determination of $\%CO_2$ on the correction or adjustment in the particulate matter concentration is a dominating factor in the overall error in the determination of PMR for low $\%CO_2$ levels. Denote C_{sa} as the adjusted value of C_s, where the relationship is

$$C_{sa} = C_s \frac{12}{\%\overline{CO}_2}, \qquad (26)$$

or the relationship in the coefficients of variation is

$$CV^2\{C_{sa}\} = CV^2\{C_s\} + CV^2\{\%CO_2\}/r, \qquad (27)$$

where r is the number of replicates used to estimate the mean, $\overline{\%CO_2}$, assuming $\sigma\{\%CO_2\} = 0.4$, then $CV\{\overline{\%CO_2}\} = 0.4/(\sqrt{r} \times \%CO_2)$ for the standard ORSAT and $CV\{\overline{\%CO_2}\} = 0.2/(\sqrt{r} \times \overline{\%CO_2})$ for the modified ORSAT. The following measures of precision are derived assuming r is taken as suggested in the Quality Assurance Document for Method 3 (ref. 2) and $m_t = 100$ mg.

$\overline{\%CO_2}$	Standard ORSAT			Modified ORSAT		
	r	CV $\{C_{sa}\}$	CV {PMR}	r	CV $\{C_{sa}\}$	CV {PMR}
2%	12	10.7%	11.0%	6	9.9%	10.2%
6%	3	9.8%	10.1%	3	9.2%	9.5%
10%	3	9.3%	9.6%	3	9.1%	9.4%

An ORSAT with 0.1 ml dimensions as depicted in figure 2 of the Operations Manual of reference 2 costs very little more than the so called standard ORSAT. Over a period of a year the difference in cost should be negligible. A cost of 200 dollars per 20 source tests is assumed for this example.

4. Minicalculator in lieu of nomographs, A4. It is assumed that the coefficient of variation of V_n/V_s is reduced in accordance with the following equation:

$$CV\{V_n/V_s|A4\} = 0.80\ CV\{V_n/V_s|A0\}\ .$$

An increase in precision and accuracy should be realized since exact values of C_p, ΔH@, and M_d would be used to determine isokinetic conditions rather than the mean values of their expected range as are now built into the nomograph. Also, routine calculation errors (i.e., error in setting and reading the nomograph) should be greatly reduced. The above assumed relationship must be considered in the analysis of the variation of m_n, C_s, and PMR as described in section 4.1.1. The corresponding coefficients of variation of PMR for the reference method and for action A4 are estimated as follows:

m_t	CV {PMR|A0}	CV {PMR|A4}
100 mg.	9.3%	8.6%
200 mg.	8.7%	8.0%
400 mg.	8.6%	7.9%

A programmable mini-calculator can be purchased for about 300 dollars. Cost of programming should be more than recovered in sampling time saved as a result of using the calculator. The increased cost is taken as 350 dollars per 20 source tests.

4.2.1 Comparison of Actions

The added cost per 20 stacks is plotted as a function of the precision of the estimated PMRs as measured by its coefficient of variation, CV{PMR} in figure 10. Examination of the plotted results enables one to quickly identify the action or combination of actions which will yield results of some desired precision. In practice it may not be reasonable to insist that the data be of a specified precision, but that the cost of reporting poor quality data is a rapidly increasing function of the data quality for data of variability exceeding a specified value. The selection of the best action option then becomes a trade-off between the overall cost and the expected precision to be achieved by its implementation. The cost of implementing an action plus the cost of reporting poor quality data are added to obtain an overall cost for the action. An assumed function of the cost of reporting poor quality data is shown as the solid curve in figure 10 as an illustration only. Its exact shape and location on the graph would have to be determined from actual cost data. Table 5

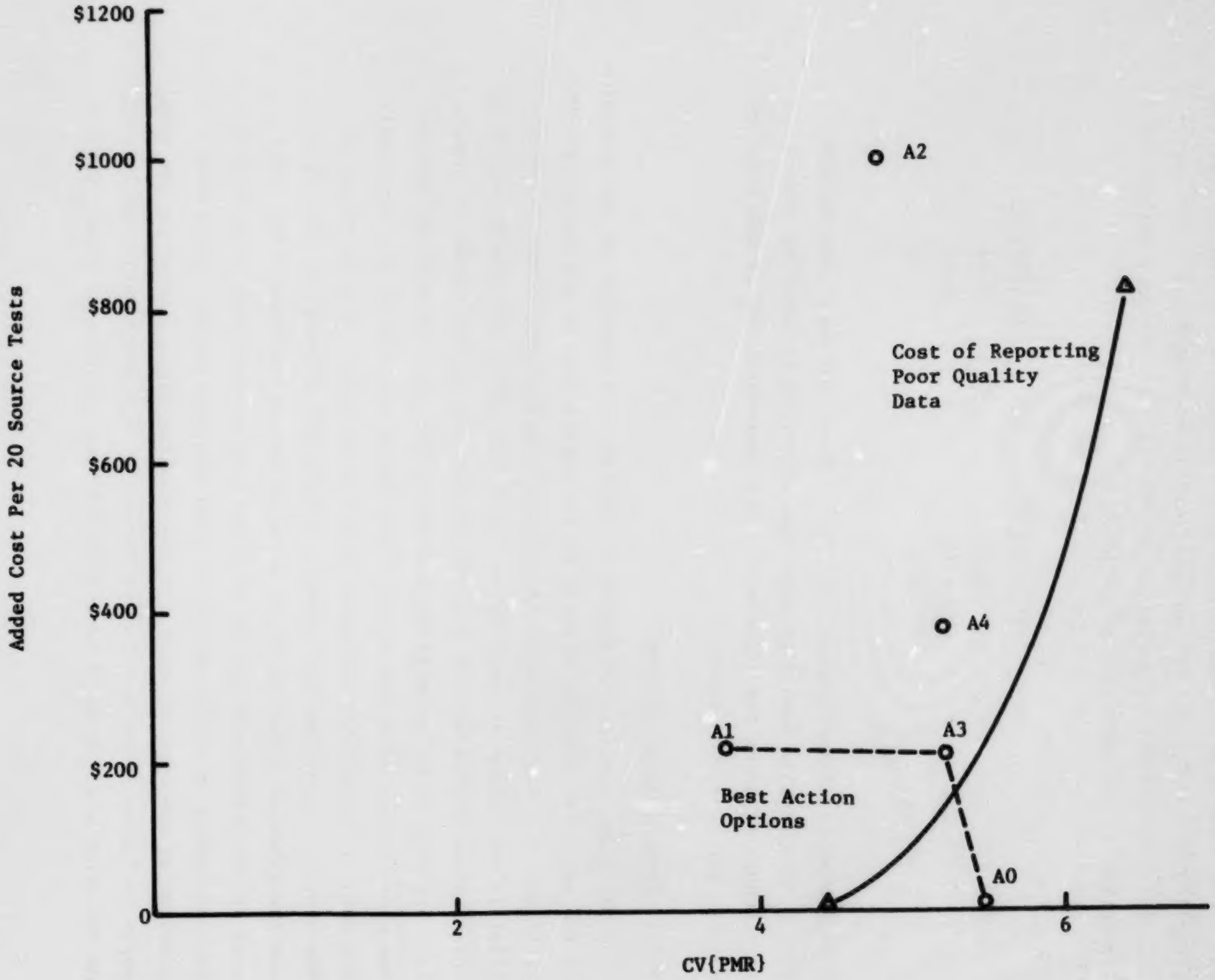


Figure 10. Added cost vs precision for selected action options for incinerator sources (M_t = 100 mg and $\%CO_2$ = 6.0).

summarizes the results of the analyses of this section which are used in plotting the data in figure 10. A comparison of the actions indicates that action A1 would be most cost-effective in improving the precision of PMR. Both actions A1 and A3 cost the same to implement, however, the larger impr vement in precision resulting from A1 shows (for this hypothetical case in figure 10) a lower overall cost since A3 shows a $400 for reporting poor quality data.

Table 5. Comparison of action options based on CV {PMR} for C_s adjusted to 12-percent CO_2 (incinerator sources)

Action option	Cost/ 20 stacks	Estimated precision (CV {PMR} in %) $m_t = 100$ mg
A0 ($\sigma\{\%CO_2\}=0.4$)	$ 0	11.0
A1	$ 200	7.6
A2	$1000	9.1
A3 ($\sigma\{\%CO_2\}=0.2$)	$ 200	9.5
A4	$ 350	10.3

4.3 PROCEDURES FOR PERFORMING A QUALITY AUDIT

"Quality audit" as used here implies a comprehensive system of planned and periodic audits to verify compliance with all aspects of the quality assurance program. Results from the quality audit provide an independent assessment of data quality. "Independent" means that the individuals performing, and as much as possible of the equipment used in the audit, are different from the regular field crew and equipment. From these data both bias and precision estimates can be made for the measurement process.

The auditor, i.e., the individual performing the audit, should have extensive background experience in source sampling, specifically with the characterization technique that he is auditing. He should be able to establish and maintain good rapport with field crews.

The functions of the auditor are summarized in the following list:

1. Observe procedures and techniques of the field team during on-site measurements.

2. Perform certain independent checks, and from previous training and experience, estimate the validity of the field crew's measurements.

3. Check/verify applicable records of equipment calibration checks and quality control charts in the field team's home laboratory.

4. Perform calculations using data obtained from the audit.

5. Compare the audit value with the field team's test value.

6. Inform the field team of the comparison results specifying any area(s) that need special attention or improvement.

7. File the records and forward the comparison results with appropriate comments to the manager.

4.3.1 Frequency of Audit

The optimum frequency of audit is a function of certain costs and desired level of confidence in the data quality assessment. A methodology for determining the optimum frequency using relevant costs is presented in subsection 4.4.4. For the costs assumed in this document, an optimum audit level of $n = 5$ for a lot size of $N = 20$ results as shown in Figure 15, page 131. However, costs will vary among field teams and types of field tests. Therefore, the most cost effective auditing level will have to be derived using local cost data according to the procedure given in subsection 4.4.4 and in the final report on this contract.

4.3.2 Collecting Audit Information

While at the sampling site, the auditor should observe the field team's overall performance of the field test. Table 6 is a sample checklist of the operations to observe. Each item on the list should be checked yes or no according to whether it was performed as recommended in the operations manual and, if applicable, the result was within specified limits. Those items checked no should be explained under comments. No checklist can cover all situations; the auditor must utilize his good

Table 6. Particulate emission determination checklist to be used by auditor

YES	NO	OPERATION
		EQUIPMENT PREPARATION AND CHECK
___	___	1. Sampling train assembled and leak checked.
___	___	2. Probe and filter box heaters checked and set for proper temparatures.
___	___	3. Stack gas temperature measuring system assembled and checked for proper operation by comparing to a mercury in glass thermometer.
___	___	4. Stack gas velocity measuring system assembled and checked for proper operation.
___	___	5. Orsat analyzer assembled and checked.
		PRELIMINARY MEASUREMENTS
___	___	6. Selection of traverse points according to Method 1.
___	___	7. Moisture content by Method 4, or equivalent.
___	___	8. Molecular weight by Method 3, or equivalent.
___	___	9. Measurement of stack dimensions.
___	___	10. Mark probe for sampling at traverse points.
		SAMPLE COLLECTION
___	___	11. Equal sampling time at each traverse point.
___	___	12. Probe temperature satisfactory throughout the test.
___	___	13. Filter box temperature 120 ± 14°C (248 ± 25°F) through the test.
___	___	14. Sample gas temperature at last impinger ≈ 20°C (68°F) throughout the test.
___	___	15. Isokinetic sampling checked and adjusted if necessary at least every 5 minutes.
___	___	16. Leak check of sampling train at end of test.
		SAMPLE RECOVERY
___	___	17. Satisfactory handling and movement of probe and filter to sample recovery area.
___	___	18. Recovery area satisfactory (i.e., space, cleanliness, etc.)

Table 6. Particulate emission determination checklist to be used by auditor (continued)

YES	NO	OPERATION
___	___	19. Sample recovery procedure adequate.
___	___	20. Proper labeling of sample containers.
___	___	21. Determination of moisture content procedure adequate.
		ANALYSIS
___	___	22. Proper equilibration of (1) filter, (2) probe wash residue, and (3) acetone blank residue.
___	___	23. Correct collected particulates for acetone blank.
___	___	24. Analytical balance checked before weighings.
		DOCUMENTATION
___	___	25. All information recorded on data sheet as obtained.
___	___	26. All unusual conditions recorded.
		COMMENTS

AUDIT MEASUREMENTS

STACK CROSS-SECTIONAL AREA, A_{s_a} ______________ m^2
DRY GAS METER CALIBRATION, γ_a ______________ Dimensionless
ORIFICE METER CALIBRATION, $\Delta H@_a$ ______________ mm H_2O
SAMPLING NOZZLE DIAMETER, D_{na} ______________ mm
SAMPLING NOZZLE CROSS-SECTIONAL AREA, A_n __________ m^2
PITOT TUBE CALIBRATION COEFFICIENT, C_{p_a} __________ Dimensionless

CALCULATIONS

1. $V_{ma} = V_m \times \gamma_a =$ ____________ m^3

2. $V_{ma(std)} = 0.3855\ V_{ma} \left(\frac{P_{bar} + \frac{\Delta H}{13.6}}{T_m} \right) =$ ___________ m^3, at standard conditions.

3. $C_{sa} = 0.001\ (m_n/V_{ma(std)}) =$ __________________ g/m^3 .

4. $Q_{sa} = 2.75 \times 10^6\ (1 - B_{ws})\ A_{sa}\ C_{p_a}\ (\sqrt{\Delta P})_{avg}\ (P_s/M_s(T_s)_{avg})^{1/2}$

   $Q_{sa} =$ ____________ m^3/hr, at standard conditions on a dry basis.

5. $PMR_a = C_{sa} \times Q_{sa} =$ ______________ g/hr.

6. $I_a = \frac{4.323\ T_s\ V_{ma(std)}}{(V_{sa})_{avg}\ A_{na}\ P_s\ \theta\ (1 - B_{ws})} =$ _____________ percent.

7. $d = \frac{\overline{PMR} - \overline{PMR}_a}{\overline{PMR}_a} \times 100 =$ ______________, percent.

Figure 11. Sample form for recording audit data

judgment and include other checks as deemed desirable for a specific situation.

A completed checklist with all yes checks implies that in the opinion of the auditor the measurement was made in such a manner that large biases resulting from poor technique are not likely to be present.

In addition to the above observations, the auditor should independently determine the stack dimensions. This should be carried out with the realization that the measurement is to be used as an estimate of the average dimension of the stack. Therefore, for example, a stack which could be out of round should have its diameter measured from as many sampling ports as possible and the average diameter used as the stack diameter in subsequent calculations. Record the cross-sectional area, A_{sa}, on the form of figure 11.

The auditor should obtain from the field team a complete set of test data, i.e., the data form of figure 4 filled in through the section on recorded test data, for all sample runs.

In the field team's home laboratory the auditor should verify, by checking calibration records and field data sheets from previous field tests, that the performance criteria as given in table 2 of section III have been satisfied over the period since the last audit. Also, using his own calibrated standards, perform the following calibration checks.

1. Using his own calibrated wet test meter, or equivalent, calibrate the dry gas meter and the orifice meter as directed in subsection 2.2.3 of section II. Record these audit values as γ_a and $\Delta H@$ in the form in figure 11.

2. Determine the sampling nozzle diameter, D_{na}, according to the procedure of subsection 2.2.1. Calculate the cross-sectional area, A_{na}, in square meters by

$$A_{na} = 0.25 \times \frac{22}{7} \times (D_{na})^2 \times \frac{1}{10^6} \frac{m^2}{mm^2}$$

$$A_{na} = 7.85 \times 10^{-7} (D_{na})^2 = \underline{\hspace{2cm}} \ m^2.$$

Record D_{na} and A_{na} on the audit data sheet of figure 11.

3. Using a calibrated pitot tube, calibrate the field pitot tube according to the procedure given in subsection 2.1.2, page 11, of the quality assurance document of this series for Method 2 (ref. 1) or the EPA revised Method 2 as reproduced in reference 4. The field pitobe assembly must be configured as shown in figure 5-1 of Appendix A. The average coefficient, C_{p_a}, applicable to the ΔP range measured in the field test is determined from this calibration data. Record C_{p_a} on the form in figure 11.

4.3.3 Treatment of Audit Data

Using the above audit data where applicable and the raw field data, perform the calculations indicated in figure 11. All variables are in the same units as used in subsection 2.5.3 of the operations manual. In figure 11 audit measurements and/or parameters computed using audit measurements are subscripted with an a. Parameters such as $(\sqrt{\Delta P})_{avg}$, $(T_s)_{avg}$, M_s, and B_{ws} should be calculated by the auditor from the field crew's original data. All calculations are recorded on the audit data sheet of figure 11.

A comprehensive discussion of estimating the true particulate mass emission rate by the average of PMR_c and PMR_a is contained in reference 23. Also, the importance of sampling isokinetically for particles down to about 5 μm in diameter is illustrated in figure 7.8, page 75, of reference 16. Unless it is known that the particle size distribution is below 5 μm for the stack gas being sampled, the above averaging technique appears to be an improvement over using just the value of PMR_c as the true emission rate.

The auditor's report of a specific field test to the manager should include copies of (1) a completed data sheet from the field team (fig. 4), (2) a completed checklist with comments (table 6), (3) a completed audit data sheet with calculations (fig. 11) and (4) a summary of the field team's strong/weak points with an overall numerical rating and recommended actions as discussed in the following subsection.

4.3.4 Overall Evaluation of Field Team Performance

In a summary-type statement, the field team should be evaluated on its overall performance. Using the checklist filled out in the field (table 6) in conjunction with the results of the comparison of audit and field team values of PMR and the circumstances under which the test was performed, the field team could be rated on a scale of 1 to 5 as follows:

5 - Excellent
4 - Above average
3 - Average
2 - Acceptable, but below average
1 - Unacceptable performance.

Justification for the rating in the form of a list of the team's strong/weak points with recommendations for improving weak points should be included in the auditor's report.

4.4 DATA QUALITY ASSESSMENT

Two aspects of data quality assessment are considered in this section. The first considers a means of estimating the precision and accuracy of the reported data, e.g., reporting the bias, if any, and the standard deviation associated with the measurements. The second consideration is that of testing the data quality against given standards using sampling by variables. For example, lower and upper limits, L and U, may be selected to include a large percentage of the measurements and outside of which it is desired to control the percentage of measurements to, say, less than 10 percent. If the data quality is not consistent with these limits, L and U, then action is taken to correct the possible deficiency before future field tests are performed and to correct the previous data when possible.

The determination of the audit level is indicated by using estimated costs associated with falsely inferring that good (poor) quality data are of poor (good) quality and with auditing n stacks. In addition, prior information concerning data quality is assumed in order to determine an expected or average cost resulting from the statistical sampling plan. The cost estimates provided herein are assumed for the purpose of illustrating the methodology. It is emphasized that managers need to supply their own costs in making such analyses.

4.4.1 Estimating the Precision/Accuracy of the Reported Data

Methods for estimating the precision (standard deviation) and bias of the particulate emission rate ($\overline{\text{PMR}}$) measurements were given in section 4.1. This section will indicate how the audit data collected in accordance with the procedure described in section 4.3 will be utilized to estimate the precision and accuracy of the measure of interest. Similar techniques can also be used by a specific firm or team to assess their own measurements. However, in this case no bias data among firms can be obtained. The audit data collected as a result of following the procedures in the previous section are the measured and audited values of $\overline{\text{PMR}}$ and the difference.

$$d_j = \frac{\overline{\text{PMR}}_j - \overline{\text{PMR}}_{aj}}{\overline{\text{PMR}}_{aj}} \times 100 \qquad (28)$$

where $\overline{\text{PMR}}_j$ = Field measured value of particulate mass emission rate, average of three replicates, and

$\overline{\text{PMR}}_{aj}$ = Audited value of particulate mass emission rate, average of three replicates.

Let the mean and standard deviation of the differences d_j, $j = 1, \ldots n$ field tests be denoted by $\bar{d}$ and s_d, respectively. Thus,

$$\bar{d} = \sum_{j=1}^{n} d_j/n \ , \qquad (29)$$

and

$$s_d = \left[\sum_{j=1}^{n} (d_j - \bar{d})^2/(n - 1)\right]^{1/2} \qquad (30)$$

4.4.2 Statistical Tests

The mean $\bar{d}$ is an estimate of the relative bias in the measurements (i.e., relative to the audited value). Assuming the audited data to be unbiased, the existence of a bias in the field data can be checked by the appropriate t-test, i.e.,

$$t_{n-1} = \frac{\bar{d} - 0}{s_d/\sqrt{n}} . \tag{31}$$

See reference 29 for a discussion of the t-test. If t is significantly large in absolute values, i.e., greater than the tabulated value of t with n - 1 degrees of freedom, which is exceeded by chance only 5 percent of the time, then the bias is considered to be real and some check should be made for a possible cause of the bias. If t is not significantly large, then the bias should be considered zero or negligible.

The standard deviation, s_d, is a function of both the standard deviation of the field measurements and of the audit measurements. Assuming the audited measurements are obtained with much greater precision than the field measurements, then the calculated s_d is an estimate of the standard deviation of the field measurements. Furthermore, since s_d is in percent, it is an estimate of the coefficient of variation, CV{PMR}. Table 7 in the following subsection contains an example calculation of $\bar{d}$ and s_d, starting with the differences for a sample size of $n = 5$.

The calculated standard deviation can then be utilized to check the reasonableness of the assumption made in section 4.1 concerning CV{PMR} = 9.3 percent for $m_t = 100$ mg, for example. (Remember that CV{PMR} is equal to s_d). The calculated standard deviation, s_d, may be directly checked against the assumed value, σ_d, by using the statistical test procedure

$$\frac{\chi^2}{f} = \frac{s_d^2}{\sigma_d^2} , \tag{32}$$

where χ^2/f is the value of a random variable having the chi-square distribution with $f = n - 1$ degrees of freedom. If χ^2/f is larger than the tabulated value exceeded only 5 percent of the time, then it would be concluded that the test procedure is yielding results with more variability than is acceptable due to some assignable cause of large variation.

The measured values should be reported along with the estimated bias, $\bar{d}$, standard deviation, s_d, the number of audits, n, and the total number of field tests, N, sample ($n \leq N$). Estimates, i.e., s_d and $\bar{d}$, which are significantly different from the assumed population parameters should be identified on the data sheet. For example, based on the data of table 7, if the field team reported a value of $\overline{PMR} = 22.9$ g/hr for one of the N field tests not audited, then that measurement would be reported as

1. Measured value, $\overline{PMR} = 22.9$ g/hr
2. Calculated bias, $\hat{\tau}\{PMR\} = \bar{d} \times \overline{PMR} = 1.51$ g/hr
3. Calculated standard deviation, $\hat{\sigma}\{\overline{PMR}\} = s_d \times \overline{PMR} = 1.9$ g/hr
4. Auditing level, $n = 5$, $N = 20$.

From the above data, users of the data can calculate confidence limits appropriate to what the data are to be used for.

The t-test and χ^2-test described above, and in further detail in the final report on this contract, are used to check on the biases and standard deviations separately. In order to check on the overall data quality as measured by the percent of measurement deviations outside prescribed limits, it is necessary to use the approach described in subsection 4.4.3 below.

4.4.3 Sampling by Variables

Because the lot size (i.e., the number of field tests performed by a team or laboratory during a particular period, normally a calendar quarter) is small, $N = 20$, and consequently, the sample size is small on the order of $n = 3$ to 7, it is important to consider a sampling by variables approach to assess the data quality with respect to prescribed limits. That is, it is desired to make as much use of the data as possible. In the variables approach, the means and standard deviations of the sample of n audits are used in making a decision concerning the data quality.

Some background concerning the assumptions and the methodology is repeated below for convenience. However, one is referred to one of a number of publications having information on sampling by variables; e.g., see references 30-33. The discussion below will be given in regard to the specific problem herein which has some unique features as compared with the usual variable sampling plans.

In the following discussion, only the audited value $\overline{\text{PMR}}$ is discussed. This same procedure can be applied to audits of sampling train components e.g., the pitot tube calibration, dry gas meter, and orifice meter.

The difference between the team-measured and audited value of $\overline{\text{PMR}}$ is designated as d_j, and the mean difference over n audits by $\bar{d}$, that is,

$$\bar{d} = \frac{100}{n} \sum_{j=1}^{n} \left(\frac{\overline{\text{PMR}}_j - \overline{\text{PMR}}_{aj}}{\overline{\text{PMR}}_{aj}} \right). \tag{33}$$

Theoretically, $\overline{\text{PMR}}_j$ and $\overline{\text{PMR}}_{aj}$ should be measures of the same particulate mass emission rate, and their difference should have a mean of zero on the average. In addition, their differences should have a standard deviation approximately equal to that associated with measurements of $\overline{\text{PMR}}_j$ separately.

Assuming three standard deviation limits (using the assumed CV = 9.3 percent as derived in the variance analysis of subsection 4.1), the values -3(9.3%) ≈ -28 percent and 3(9.3%) ≈ 28 percent define lower and upper limits, L and U, respectively, outside of which it is desired to control the proportion of differences, d_j. Following the method given in reference 30, a procedure for applying the variables sampling plan is described below. Figures 12 and 13 illustrate examples of satisfactory and unsatisfactory data quality with respect to the prescribed limits L and U.

The variables sampling plan requires the sample mean difference, $\bar{d}$; the standard deviation of these differences, s_d; and a constant, k, which is determined by the value of p, the proportion of the differences outside the limits of L and U. For example, if it is desired to control at 0.10 the probability of not detecting lots with data quality p equal to 0.10 (or 10% of the individual differences outside L and U) and if the sample size n = 5, then the value of k can be obtained from Table II of reference

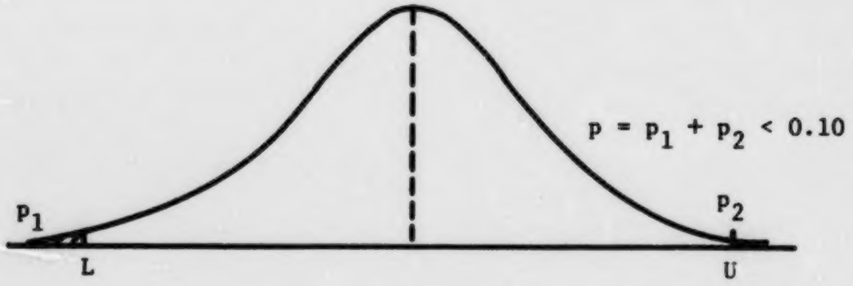

$p = p_1 + p_2 < 0.10$
p_1
p_2
L
U

Figure 12. Example illustrating $p < 0.10$ and satisfactory data quality.

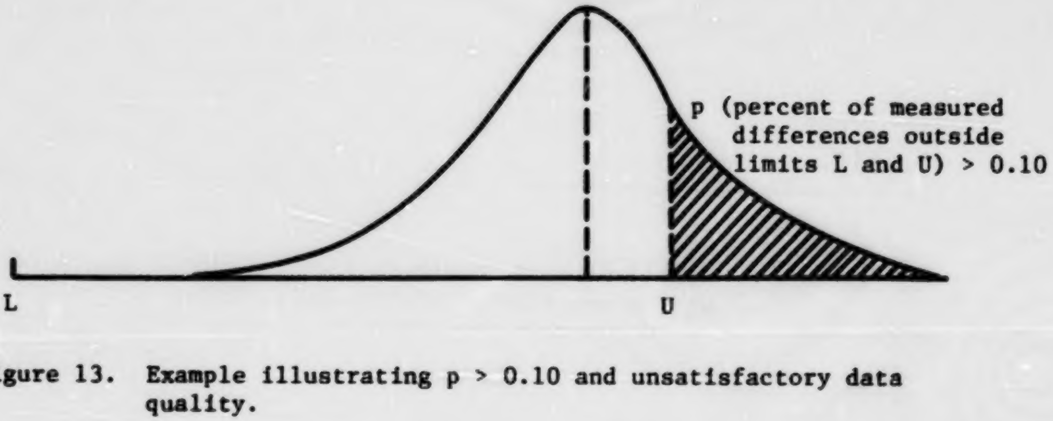

p (percent of measured differences outside limits L and U) > 0.10
L
U

Figure 13. Example illustrating $p > 0.10$ and unsatisfactory data quality.

Table 7. Computation of mean difference, $\bar{d}$, and standard deviation of differences, s_d

General formulas		Specific example	
$d_j = \frac{\overline{PMR}_j - \overline{PMR}_{aj}}{\overline{PMR}_{aj}} \times 100$		Data (percent)	
d_1	d_1^2	12.0	14.4
d_2	d_2^2	-6.0	3.6
d_3	d_3^2	3.0	0.9
d_4	d_4^2	15.0	22.5
d_5	d_5^2	9.0	8.1
Σd_j	Σd^2_j	33.0	49.5
$\bar{d} = \frac{\Sigma d_j}{n}$		$\bar{d} = 6.6\%$	
$s_d^2 = \frac{\Sigma d_j^{\,2} - (\Sigma d_j)^2/n}{(n - 1)}$		$s_d^2 = 69.3\%$	
$s_d = \sqrt{s_d^2}$		$s_d = 8.3\%$	

Table 8. Sample plan constants, k for P{not detecting a lot with proportion p outside limits L and U} ≤ 0.1

Sample size n	k(p = 0.2)	k(p = 0.1)
3	3.039	4.258
5	1.976	2.742
7	1.721	2.334
10	1.595	2.112
12	1.550	2.045

30. The values of $\bar{d}$ and s_d are computed in the usual manner; see table 8 for formulas and a specific example. Given the above information, the test procedure is applied and subsequent action is taken in accordance with the following criteria:

1. If both of the following conditions are satisfied:

$$\bar{d} - k\ s_d \geq L = - -28 \text{ percent,} \tag{34}$$

$$\bar{d} + k\ s_d \leq U = 28 \text{ percent} \tag{35}$$

the individual differences are considered to be consistent with the deficiencies exist in the measurement process as carried out for that particular lot (group) of field tests. These deficiencies should be identified and corrected before future field tests are performed. Data corrections should be made when possible, i.e., if a quantitative basis is determined for correction.

Table 8 contains a few selected values of n, p, and k for convenient reference.

Using the values of $\bar{d}$ and s_d in table 8, $k = 2.742$ for a sample size $n = 5$, and $p = 0.10$, the test criteria can be checked; i.e.,

$$\bar{d} - k\ s_d = 6.6 - 2.742\ (8.3) = -16.2 > L = -28 \text{ percent} \tag{36}$$

and

$$\bar{d} + k\ s_d = 6.6 + 2.742(8.3) = 29.4 > U = 28 \text{ percent.} \tag{37}$$

Therefore, the upper limit is not satisfied and the lot of $N = 20$ measurements is not consistent with the prescribed quality limits. The plan is designed to protect against not detecting lots with 10 percent or more defects (deviations falling outside the designated limits L and U) with a risk of 0.10; that is , on the average, 90 percent of the lots with 10 percent or more defects will be detected by this sampling plan.

4.4.4 Cost Versus Audit Level

The determination of the audit level (sample size n) to be used in assessing the data quality with reference to prescribed limits L and U can be made on a statistical basis by defining acceptable risks for type I and type II errors, knowing or estimating the quality of the incoming data, and specifying the described level of confidence in the reported data, or on a cost bassis as described herein. In this section, cost data associated with the audit procedure are estimated or assumed for the purpose of illustrating a method of approach and identifying which costs should be considered.

A model of the audit process, associated costs, and assumptions made in the determination of the audit level is provided in figure 14. It is assumed that a collection of source emissions tests for N stacks is to be made by a particular firm, and that n measurements ($n \leq N$) are to be audited at a cost, $C_A = b + cn$, where b is a constant independent for n, and c is the cost perstack measurement audited. In order to make a specific determination of n, it is also necessary to make some assumptions about the quality of the source emissions data from several firms. For example, it is assumed in this analysis that 50 percent of the data lots are of good quality, i.e., one half of the firms are adhering to good data quality assurance practice, and that 50 percent of the data lots are of poor quality. Based on the analysis in section 4.1, good quality data is defined as that which is consistent with the estimated precision/bias using the reference method. Thus, if the data quality limits L and U are taken to be the lower and upper 3σ limits corresponding to limits used in a control chart, the quality of data provided by a firm adhering to the recommended quality assurance procedures should at most contain about 0.3 percent defective measurements (i.e., outside the limits defined by L and U). Herein, good quality data is defined as that containing at most 10 percent defective measurements. The definition of poor quality data is somewhat arbitrary, for this illustration it is taken as 25 percent outside L and U.

In this audit procedure the data are declared to be of acceptable quality if both of the following inequalities are satisfied

$$\bar{d} + ks_d < U \qquad (39)$$

$$\bar{d} - ks_d > L, \tag{40}$$

where $\bar{d}$ and s_d are the mean and standard deviation of the data quality characteristic (i.e., the difference of the field and audited measurements) being checked, and not of desired quality if one or both inequalities is violated, as described in section 4.3. The cost associated with these actions are assumed to be as follows:

C_A = Audit cost = b + cn. b is assumed to be zero for this example and c is taken as $600/measurement.

$C_{P|G}$ = Cost of falsely inferring that the data are of poor quality, P, given that the data are of good quality, G. This cost is assumed to be one-half the cost of collecting emissions data for N = 20 stacks (i.e., 0.5 × $1000 × 20 = $10,000). This cost would include that of searching for an assignable cause of the inferred data deficiency when none exists, partial repetition of data collection, decision resulting in the purchase of equipment to reduce emission levels of specific pollutants, etc.

$C_{G|P}$ = Cost of falsely stating that the data are of good quality, G, given that they are of poor quality. This cost is assumed to be $15,000 (= 0.75 × $1,000 × 20). This cost is associated with health effects, litigation, etc.

$C_{P|P}$ = Cost savings resulting from correct identification of poor quality data. This cost is taken to be $7,500, i.e., equal to one-half of $C_{P|G}$ or equal to 0.375 × $1,000 × 20, the total cost of data collection.

These costs are given in figure 14. These cost data are then used in conjunction with the a priori information concerning the data quality to select an audit level n. Actually, the audit procedure requires the selection of the limits L and U, n, and k. L and U are determined on the basis of the analysis of section 4.1. The value of k is taken to be the value associated with n in table 8 of section 4.4.3, i.e., the value selected on a statistical basis to control the percentage of data outside

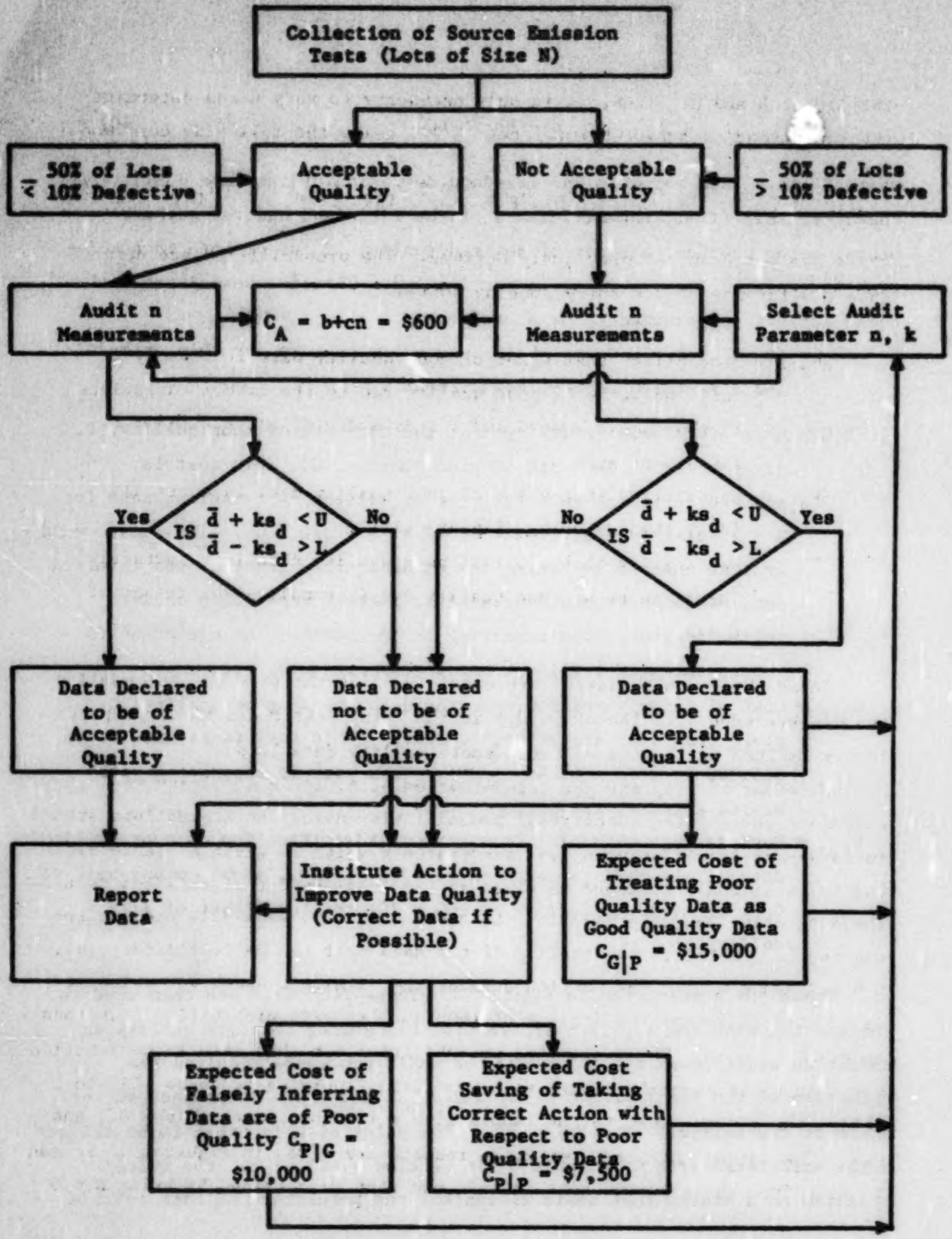

Collection of Source Emission Tests (Lots of Size N)
50% of Lots ≤ 10% Defective
Acceptable Quality
Not Acceptable Quality
50% of Lots > 10% Defective
Audit n Measurements
$C_A = b+cn = \$600$
Audit n Measurements
Select Audit Parameter n, k
Yes
IS $\bar{d} + ks_d < U$ $\bar{d} - ks_d > L$
No
No
IS $\bar{d} + ks_d < U$ $\bar{d} - ks_d > L$
Yes
Data Declared to be of Acceptable Quality
Data Declared not to be of Acceptable Quality
Data Declared to be of Acceptable Quality
Report Data
Institute Action to Improve Data Quality (Correct Data if Possible)
Expected Cost of Treating Poor Quality Data as Good Quality Data $C_{G|P} = \$15,000$
Expected Cost of Falsely Inferring Data are of Poor Quality $C_{P|G} = \$10,000$
Expected Cost Saving of Taking Correct Action with Respect to Poor Quality Data $C_{P|P} = \$7,500$

Figure 14. Flow chart of the audit level selection process.

the limits L and U. Thus, it is only necessary to vary n and determine the corresponding expected total cost E(TC) using the following cost model.

$$E(TC) = - C_A - 0.5\ P_{P|G}\ C_{P|G} + 0.5\ P_{P|P}\ C_{P|P} - 0.5\ P_{G|P}\ C_{G|P} \quad (41)$$

where the costs are as previously defined. The probabilities are defined in a similar way to the corresponding costs.

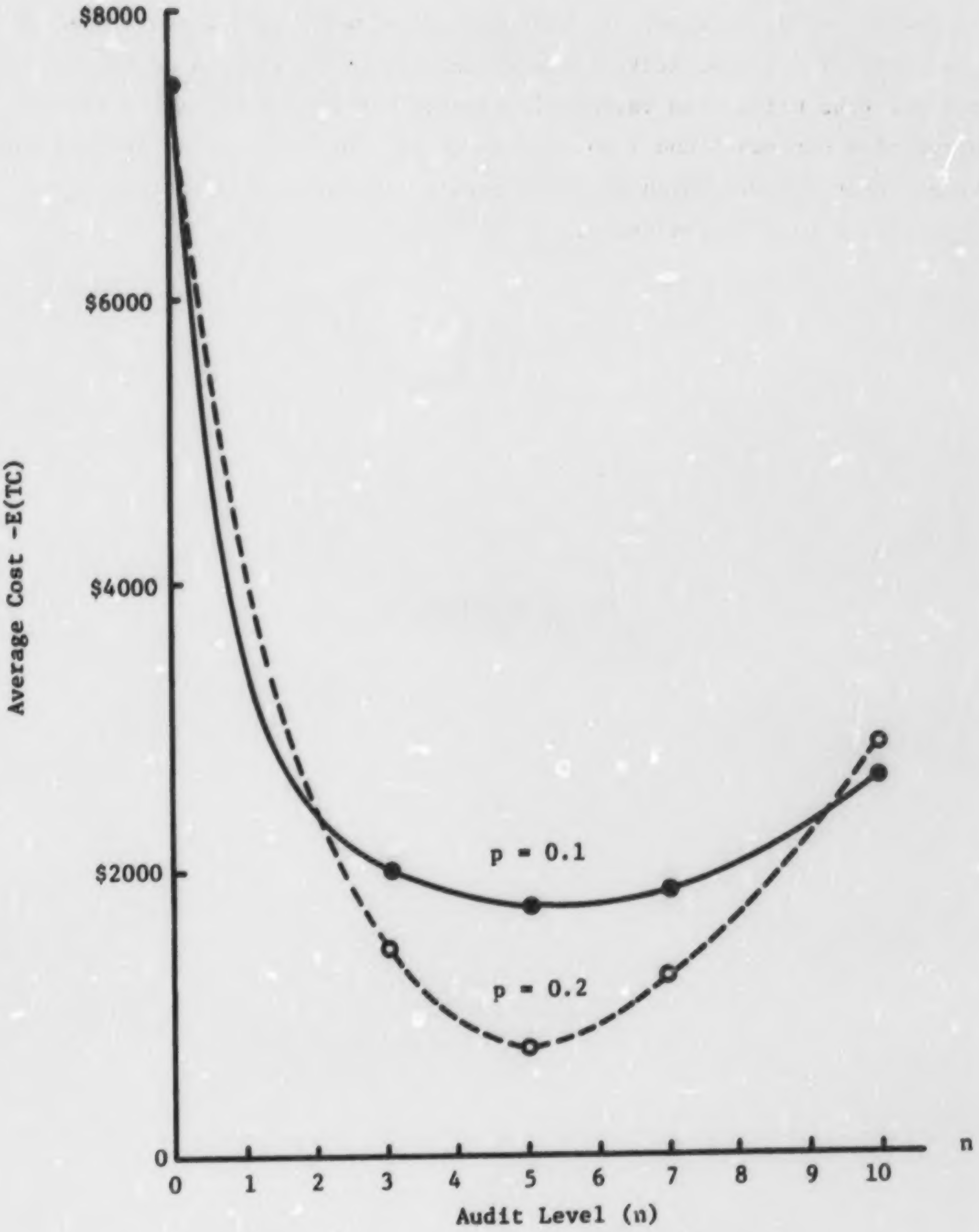
$P_{P|G}$ = Probability that a lot of good quality data is falsely inferred to be of poor quality due to the random variations in the sample mean $\bar{d}$ and standard deviation, s_d, in small samples of size n.

$P_{P|P}$ = Probability that a lot of poor quality data is correctly identified as being of poor quality.

$P_{G|P}$ = Probability that a lot of poor quality data is incorrectly judged to be of good quality due to sampling variations of $\bar{d}$ and s.

These three probabilities are conditional on the presumed lot quality and are preceded by a factor of 0.5 in the total cost model to correspond to the assumed percentage of good (poor) quality data lots.

In order to complete the determination of n, it is necessary to calculate each of the conditional probabilities using the assumptions stated for a series of values of n (and associated k which is given in table 9). The computational procedure is given in the Final Report of this contract. These calculations were made for the cases n = 3, 5, 7, and 10 and for two degrees of control on the quality of the data that can be tolerated, i.e., p = 0.2 and p = 0.1, the portion outside the limits L and U for which it is desired to accept the data as of good quality with probability less than or equal to 0.10. These computed probabilities are then used in conjunction with the costs associated with each condition, applying equation (41) to obtain the average cost versus sample size n for the two cases p = 0.1 and 0.2. The curves obtained from these results are given in figure 15. It can be seen from these curves that the minimum cost is obtained by using n = 5 independent of p. However, it must be recognized that the costs used in the example are for illustrative purposes and may vary from one region to another, thus within the reasonable uncertainty of the estimated costs, values of n between 3 and 7 would seem to be reasonable. The assumed costs suggest that p = 0.2 which tends to permit data of poorer quality to be accepted, is more cost effective.



p = Proportion defective measurements in the "lot"

P{Acc. lot with p} ≤ 0.1

Figure 15. Average cost vs audit level (n).

LIST OF REFERENCES

1. EPA-650/14-74-005-a "Guidelines for Development of a Quality Assurance Program--Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)" Environmental Protection Agency, Research Triangle Park, North Carolina 27711, 1974.

2. EPA-650/14-74-005-b "Guidelines for Development of a Quality Assurance Program--Gas Analysis for Carbon Dioxide, Oxygen, Excess Air, and Dry Molecular Weight" Environmental Protection Agency, Research Triangle Park, North Carolina 27711, 1974.

3. EPA-650/14-74-005-c "Guidelines for Development of a Quality Assurance Program--Determination of Moisture in Stack Gases." Environmental Protection Agency, Research Triangle Park, North Carolina 27711, 1974.

4. F. Smith _et al_. "Guidelines for Development of Quality Assurance Programs Applicable to Stationary Source Emission Stream Characterization Techniques," Final Report, EPA Contract No. 68-02-1234, Research Triangle Institute, Research Triangle Park, North Carolina 27709, 1976.

5. E. S. Kipecki, "Stainless Steels." _Machine Design_, 1970 Metals Reference Issue 4, Vol. 42, Cleveland, Ohio: Penton Pbulishing Company, February 12, 1970, pp. 34-38.

6. R. M. Martin. _Construction Details of Isokinetic Source Sampling Equipment_. Publ. No. APTD-0581. Air Pollution Control Office, Environmental Protection Agency, Research Triangle Park, North Carolina 27711, 1971.

7. A. W. Gnyp _et al_. "An Experimental Investigation of the Effect of Pitot Tube-Sampling Probe Configurations on the Magnitude of the S-Type Pitot Tube Coefficient for Commercially Available Source Sampling Probes," Technology Development and Appraisals Section, Air Resources Branch, Ministry of the Environment, Province of Ontario, Toronto, Canada; University of Windsor, February, 1975.

8. W. S. Smith. _Stack Sampling News_ 1, No. 7, 1974.

9. J. J. Rom. _Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment_. Publ. No. APTD-0576. Office of Air Programs, Environmental Protection Agency, Research Triangle Park, North Carolina, 1972.

10. "Occupational Safety and Health Standards: National Concensus Standards and Established Federal Standards." _Federal Register_ 36, No. 105, May 29, 1971.

11. "Standards of Performance for New Sources." _Federal Register_ 36, No. 247, December 23, 1971.

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12. W. S. Smith and D. J. Grose. *Stack Sampling Nomographs for Field Estimations*. Entropy Environmentalists, Inc., Research Triangle Park, North Carolina, 1973.

13. W. S. Smith. *Stack Sampling News* 1, No. 1, 1973.

14. R. F. Yarnor. *Industrial Source Sampling*. Ann Arbor Science Publishers, Inc., 1971.

15. W. J. Mitchell. *Evaluation Report: Additional Studies on Obtaining Replicate Particulate Samples from Stationary Sources*. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, 1973.

16. EPA-650/4-74-029 "Collaborative Study of Method for the Determination of Particulate Matter Emissions from Stationary Sources (Portland Cement Plant)," Environmental Protection Agency, Research Triangle Park, North Carolina 27711, May 1974.

17. EPA-650/4-74-021 "Collaborative Study of Method for the Determination of Particulate Matter Emissions from Stationary Sources (Fossil Fuel-Fired Steam Generators)," Environmental Protection Agency, Research Triangle Park, North Carolina 27711, June, 1974.

18. EPA-650/4-74-022 "Collaborative Study of Method for the Determination of Particulate Matter Emissions from Stationary Sources (Municipal Incinerators), Environmental Protection Agency, Research Triangle Park, North Carolina 27711, July, 1974.

19. *Administrative and Technical Aspects of Source Sampling for Particulates*. Publ. No. APTD-1289. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, May, 1971.

20. E. L. Grant and R. S. Leavenworth. *Statistical Quality Control*. 4th ed., St. Louis: McGraw-Hill, 1972.

21. D. A. Simons. *Practical Quality Control*. Reading, Mass.: Addison-Wesley Publishing Company, 1970, pp. 131-150.

22. EPA-600/9-76-005 "Quality Assurance Handbook for Air Pollution Measurement Systems," Volume-1 Principles, Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Research Triangle Park, North Carolina 27711, March, 1976.

23. P. K. Mueller *et al*. "Selection of Filter Media: An Annotated Outline." Presented at the 13th Conference on Methods in Air Pollution and Industrial Hygiene Studies, University of California, Berkeley, California, October 30-31, 1972.

24. R. M. Burton *et al*. "Field Evaluation of the High-Volume Particle Fractionating Cascade Impactor--A Technique for Respirable Sampling."

Presented at the 65th Annual Meeting of the Air Pollution Control Association, June 18-22, 1972.

25. W. C. L. Hemeon and A. W. Black. "Stack Dust Sampling: In Stack Filter or EPA Train." Journal of the Air Pollution Control Association 22, No. 7, 1972, pp. 516-518.

26. W. S. Smith. "A Matter of Definition." Stack Sampling News 1, No. 1, Technomic Publishing Co., Inc. Westport, Conn., July, 1973.

27. B. D. Bloomfield. "Source Testing." Air Pollution, Volume II, 2nd edition, A. C. Stern, ed. New York: Academic Press, 1968, Chapter 28.

28. W. S. Smith et al. "A Method of Interpreting Stack Sampling Data." Stack Sampling News 1, No. 2, 1974, pp. 8-17.

29. A. Hald. Statistical Theory with Engineering Applications. New York: John Wiley and Sons, 1952.

30. D. B. Owen. "Variables Sampling Plans Based on the Normal Distribution." Technometrics 9, No. 3, August, 1967.

31. D. B. Owen. "Summary of Recent Work on Variables Acceptance Sampling with Emphasis on Non-normality." Technometrics 11, 1969, pp. 631-637.

32. K. Takogi. "On Designing Unknown Sigma Sampling Plans Based on a Wide Class on Non-normal Distributions." Technometrics 14, 1972.

33. C. Eisenhart, M. Hastay, and W. A. Wallis, eds. Techniques of Statistical Analysis. Statistical Research Group, Columbia Univ. New York: McGraw-Hill, 1947.

METHOD 5--DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES

1. Principle and Applicability

1.1 Principle. Particulate matter is withdrawn isokinetically from the source and collected on glass fiber filter maintained at temperatures equal to or less than 120 ± 14° C (248 ± 25° F) or such other temperature as specified by an applicable subpart of the standards. The particulate mass is determined gravimetrically after removal of uncombined water.

1.2 Applicability. This method is applicable for the determination of particulate emissions from stationary sources only when specified by the test procedures for determining compliance with new source performance standards.

2. Apparatus

2.1 Sampling train. A schematic of the sampling train used in this method is shown in Figure 5-1. Commercial models of this train are available. However, if one desires to build his own, complete construction details are described in APTD-0581; for changes from the APTD-0581 document and for allowable modifications to Figure 5-1, see the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576. Since correct usage is important in obtaining valid results, all users should read the APTD-0576 document and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein.

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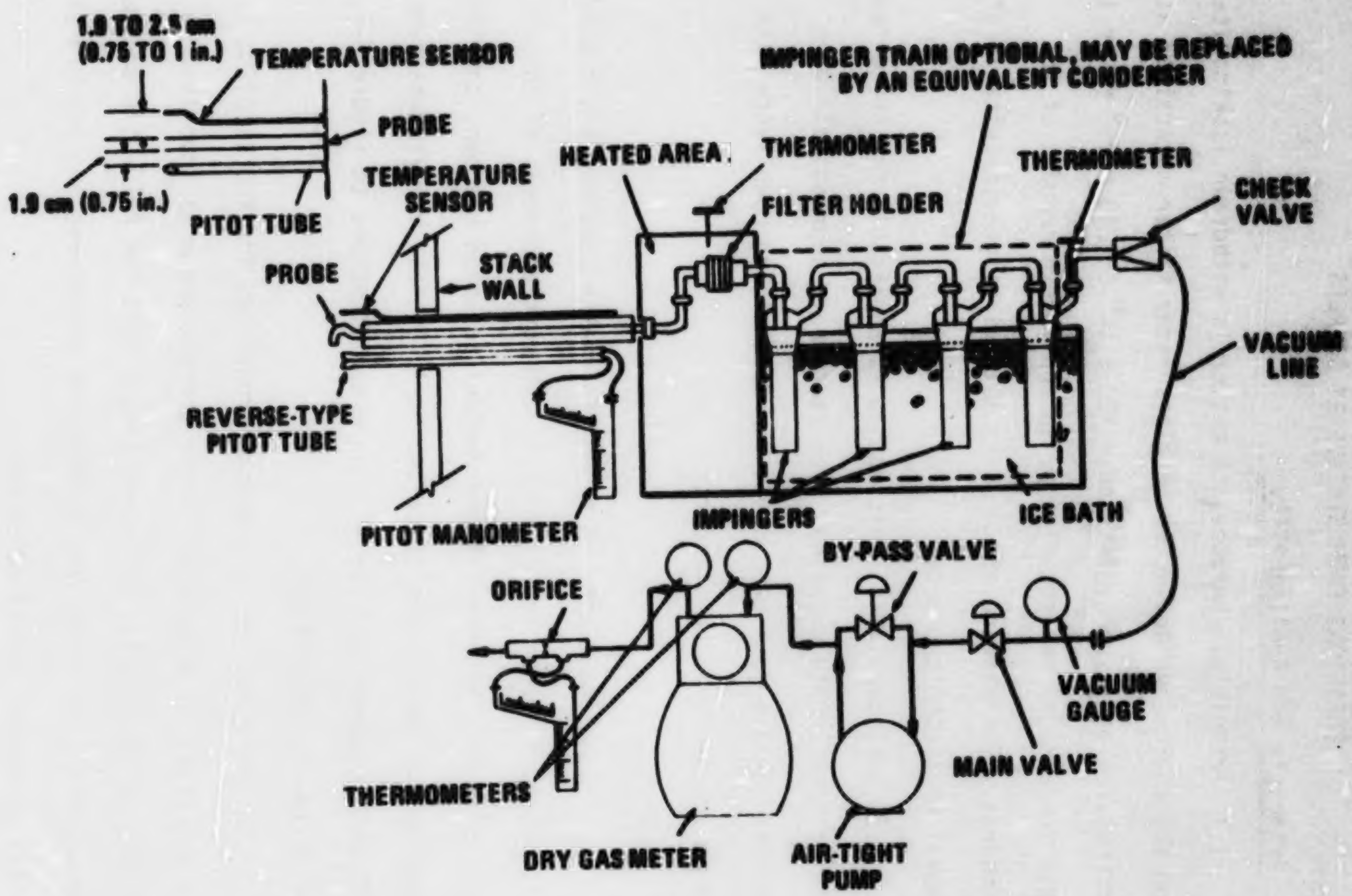
1.9 TO 2.5 cm
(0.75 TO 1 in.)
TEMPERATURE SENSOR
PROBE
1.9 cm (0.75 in.)
PITOT TUBE
TEMPERATURE SENSOR
PROBE
STACK WALL
REVERSE-TYPE PITOT TUBE
PITOT MANOMETER
HEATED AREA
THERMOMETER
FILTER HOLDER
IMPINGER TRAIN OPTIONAL, MAY BE REPLACED BY AN EQUIVALENT CONDENSER
THERMOMETER
CHECK VALVE
VACUUM LINE
IMPINGERS
ICE BATH
BY-PASS VALVE
ORIFICE
THERMOMETERS
DRY GAS METER
AIR-TIGHT PUMP
MAIN VALVE
VACUUM GAUGE

Figure 5-1. Particulate-sampling train.

2.1.1 Probe nozzle--Stainless steel (316) with sharp, tapered leading edge. The angle of taper shall be ≤ 30° and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. The wall thickness of the nozzle shall be less than or equal to that of a 20 gauge tubing, i.e., 0.165 cm (0.065 in.) and the distance from the tip of the nozzle to the first bend or point of disturbance shall be at least two times the outside nozzle diameter. The nozzle shall be constructed from seamless stainless steel tubing. Other configurations and construction material may be used with approval from the Administrator.

A range of sizes suitable for isokinetic sampling should be available, e.g., 0.32 cm (1/8 in.) up to 1.27 cm (1/2 in.) (or larger if higher volume sampling trains are used) inside diameter (ID) nozzles in increments of 0.16 cm (1/16 in.). Each nozzle shall be calibrated according to the procedures outlined in the calibration section.

2.1.2 Probe liner--Borosilicate or quartz glass tubing with a heating system capable of maintaining a gas temperature at the exit end during sampling of no greater than 120 ± 14° C (248 ± 25° F) or no greater than such other temperature as specified by an applicable subpart of the standards. Since the actual temperature at the outlet of the probe is not monitored during sampling, probes constructed according to APTD-0581 and utilizing the calibration curves of APTD-0576 or calibrated according to the procedure outlined in APTD-0576 will be considered as acceptable.

Borosilicate or quartz glass probe liners shall be used for temperatures up to about 480° C (900° F) and quartz liners for temperatures up to about 900° C (1650° F). Both may be used at higher temperatures for short periods of time, but must be approved by the Administrator. The softening temperature for borosilicate is 820° C (1508° F) and for quartz it is 1500° C (2732° F).

When length limitations, i.e. greater than about 2.5 m (8.2 ft), are encountered at temperatures less than 320° C (608° F), stainless steel (316) or Incoloy 825[1] (both of seamless tubing), or other materials as approved by the Administrator, may be used. Metal probes for sampling gas streams at temperatures in excess of 320° C (608° F) must be approved by the Administrator.

2.1.3 Pitot tube--Type S, or other device approved by the Administrator, attached to probe to allow constant monitoring of the stack gas velocity. The face openings of the pitot tube and the probe nozzle shall be adjacent and parallel to each other, not necessarily on the same plane, during sampling. The free space between the nozzle and pitot tube shall be at least 1.9 cm (0.75 in.). The free space shall be set based on a 1.3 cm (0.5 in.) ID nozzle. If the sampling train is designed for sampling at higher flow rates than that described in APTD-0581, thus necessitating the use of larger sized nozzles, the largest sized nozzle shall be used to set the free space.

The pitot tube must also meet the criteria specified in Method 2 and calibrated according to the procedure in the calibration section of that method.

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[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

2.1.4 Differential pressure gauge--Inclined manometer capable of measuring velocity head to within 10% of the minimum measured value. Below a differential pressure of 1.3 mm (0.05 in.) water gauge, micromanometers with sensitivities of 0.013 mm (0.0005 in.) should be used. However, micromanometers are not easily adaptable to field conditions and are not easy to use with pulsating flow. Thus, methods or other devices acceptable to the Administrator may be used when conditions warrant.

2.1.5 Filter holder--Borosilicate glass with a glass frit filter support and a silicone rubber gasket. Other materials of construction may be used with approval from the Administrator, e.g. if probe liner is stainless steel, then filter holder may be stainless steel. The holder design shall provide a positive seal against leakage from the outside or around the filter.

2.1.6 Filter heating system--Any heating system capable of maintaining a temperature around the filter holder during sampling of no greater than 120 ± 14° C (248 ± 25° F), or such other temperature as specified by an applicable subpart of the standards. A temperature gauge capable of measuring temperature to within 3° C (5.4° F) shall be installed such that temperature around the filter holder can be regulated and monitored during sampling. Heating systems other than shown in APTD-0581 may be used.

2.1.7 Condenser--Any system that cools the sample gas stream and allows measurement of the water condensed and moisture leaving the condenser, each to within 1 ml or 1 g. Acceptable means are to measure the condensed water either gravimetrically or volumetrically and to measure the moisture leaving the condenser by (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law or (2) by passing the sample gas stream through a tared silica gel trap with exit gases kept below 20^{0} C (68^{0} F) and determining the weight gain.

Note: If "condensible particulate matter" is desired, in addition to moisture content, the following system shall be used--four impingers connected in series with ground glass, leak free fittings or any similarly leak free noncontaminating fittings. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with a 1.3 cm (1/2 in.) ID glass tube extending to about 1.3 cm (1/2 in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. Individual States or control agencies requiring this information shall be contacted as to the sample recovery and analysis of the impinger contents.

For purposes of writing the procedure of this method, the system described in the note above will be used for determining the moisture content of the stack gas. Modifications (e.g. using flexible connections between the impingers or using materials other than glass) may be used with approval from the Administrator.

If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel still be used between the condenser system and pump to prevent moisture condensation in the pump and metering devices.

Unless otherwise specified by the Administrator, flexible vacuum lines may be used to connect the filter holder to the condenser.

2.1.8 Metering system--Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3° C (5.4° F), dry gas meter with 2% accuracy, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume. Sampling trains utilizing metering systems designed for higher flow rates than that described in APTD-0581 or APTD-0576 may be used provided that the specifications in section 2 of this method are met. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

2.1.9 Barometer--Mercury, aneroid, or other barometers capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby weather bureau station, in which case the station value shall be requested and an adjustment for elevation differences shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase.

2.1.10 Gas density determination equipment--Temperature and pressure gauges and gas analyzer as described in Methods 2 and 3.

2.1.11 Temperature and pressure gauges--If Dalton's law is used, to monitor temperature and pressure at condenser outlet. The temperature gauge shall have an accuracy of 1° C (2° F). The pressure gauge shall be capable of measuring pressure to within 2.5 mm Hg

(0.1 in. Hg). If silica gel is used in the condenser system the temperature and pressure must be measured before the silica gel component.

2.2 Sample recovery.

2.2.1 Probe liner and probe nozzle brushes--Nylon bristles with stainless steel wire handles. The probe brush shall have extensions, at least as long as the probe, of stainless steel, nylon, teflon, or similarly inert material. Both brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

2.2.2 Glass wash bottles--Two.

2.2.3 Glass sample storage containers--Chemically resistant, borosilicate narrow mouth glass bottles, for acetone washes, 500 ml or 1,000 ml. Screw cap closures shall be teflon rubber-backed liners or of such construction so as to be leak free and prevent chemical attack from the acetone. Other types of containers must be approved by the Administrator.

2.2.4 Petri dishes--For filter samples, glass or plastic, unless otherwise specified by the Administrator.

2.2.5 Graduated cylinder and/or balance--To measure condensed water to within 1 ml or 1 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. Any of these balances are suitable for use here and in section 2.3.4.

2.2.6 Plastic storage containers--Air tight containers to store silica gel.

2.2.7 Funnel and rubber policeman--To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

2.3 Analysis.

2.3.1 Glass weighing dishes.

2.3.2 Desiccator.

2.3.3 Analytical balance--To measure to within 0.1 mg.

2.3.4 Balance--To measure to within 0.5 g.

2.3.5 Beakers--250 ml.

2.3.6 Hygrometer--To measure the relative humidity of the laboratory environment.

2.3.7 Temperature gauge--To measure the temperature of the laboratory environment.

3. Reagents

3.1 Sampling.

3.1.1 Filters--Glass fiber filters, without organic binder exhibiting at least 99.95% efficiency (≤ 0.05% penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM standard method D 2986-71. Test data from the supplier's quality control program is sufficient for this purpose.

3.1.2 Silica gel--Indicating type, 6-16 mesh. If previously used, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received.

3.1.3 Water--When analysis of the material caught in the impingers is required, distilled water shall be used. Run blanks prior to field use to eliminate a high blank on test samples.

3.1.4 Crushed ice.

3.1.5 Stopcock grease--Acetone insoluble, heat stable silicone grease. This is not necessary if screw-on connectors with teflon sleeves, or similar, are used.

3.2 Sample recovery.

3.2.1 Acetone--Reagent grade, ≤ 0.001% residue, in glass bottles. Acetone from metal containers generally has a high residue blank and should not be used. Sometimes, suppliers transfer acetone to glass bottles from metal containers. Thus, acetone blanks shall be run prior to field use and only acetone with low blank values (≤ 0.001%) shall be used.

3.3 Analysis.

3.3.1 Acetone--Same as 3.2.1.

3.3.2 Desiccant--Anhydrous calcium sulfate, indicating type.

4. Procedure

4.1 Sampling. The sampling shall be conducted by competent personnel experienced with this test procedure.

4.1.1 Pretest preparation. All the components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified herein.

Weigh approximately 200-300 g of silica gel in air tight containers to the nearest 0.5 g. Record the total weight, both silica gel and container, on the container. More silica gel may be used but care should be taken during sampling that it is not entrained and carried out from the impinger. As an alternative, the silica gel may be weighed directly in the impinger or its sampling holder just prior to the train assembly.

Check filters visually against light for irregularities and flaws or pinhole leaks. Label a filter of proper diameter on the back side near the edge using numbering machine ink. As an alternative, label the shipping container (glass or plastic petri dishes) and keep the filter in this container at all times except during sampling and weighing.

Desiccate the filters at 20 ± 5.6° C (68 ± 10° F) and ambient pressure for at least 24 hours and weigh at 6 or more hour intervals to a constant weight, i.e., $\leq$ 0.5 mg change from previous weighing, and record results to the nearest 0.1 mg. During each weighing the filter must not be exposed to the laboratory atmosphere for a period greater than 2 minutes and a relative humidity above 50%.

4.1.2 Preliminary determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2 and moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate calculations. Estimates may be used. However, final results will be based on actual measurements made during the test.

Select a nozzle size based on the range of velocity heads such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the differential pressure gauge is capable of measuring the minimum velocity head value to within 10%, or as specified by the Administrator.

Select a suitable probe liner and probe length such that all traverse points can be sampled. Consider sampling from opposite sides for large stacks to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that the sampling time per point is not less than 2 min. or some greater time interval as specified by the Administrator and the sample volume that will be taken will exceed the required minimum total gas sample volume specified in the test procedures for the specific industry. The latter is based on an approximate average sampling rate. Note also that the minimum total sample volume is corrected to standard conditions.

It is recommended that 1/2 or an integral number of minutes be sampled at each point in order to avoid timekeeping errors.

In some circumstances, e.g. batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

4.1.3 Preparation of collection train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Place 100 ml of water in each of the first two impingers, leave the third impinger empty, and place approximately 200-300 g or more, if necessary, of preweighed silica gel in the fourth impinger. Record the weight of the silica gel and container to the nearest 0.5 g. Place the container in a clean place for later use in the sample recovery.

Using a tweezer or clean disposable surgical gloves, place the labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to not allow the sample gas stream to circumvent the filter. Check filter for tears after assembly is completed.

When glass liners are used, install selected nozzle using a Viton A O-ring when stack temperatures are less than 260° C (500° F) or an asbestos string gasket when temperatures are higher. The Viton A O-ring and asbestos string gasket are installed as a seal where the nozzle is connected to a glass liner. See APTD-0576 for details. When metal liners are used, install the nozzle as above or by a leak free direct mechanical connection. Mark probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Unless otherwise specified by the Administrator, attach a temperature probe to the metal sheath of the sampling probe so that the sensor extends beyond the probe tip and does not touch any metal. Its position should be about 1.9 to 2.54 cm (0.75 to 1 in.) from the pitot tube and probe nozzle to avoid interference with the gas flow.

Set up the train as in Figure 5-1, using, if necessary, a very light coat of silicone grease on all ground glass joints, greasing only the outer portion (see APTD-0576) to avoid possibility of contamination by the silicone grease. With approval from the Administrator, a glass cyclone may be used between the probe and filter holder.

Place crushed ice around the impingers.

4.1.4 Leak check procedure--After the sampling train has been assembled, turn on and set the filter and probe heating system to the power required to reach a temperature of 120 ± 14° C (248 ± 25° F) or such other temperature as specified by an applicable subpart of the standards for the leak check. (If water condensation is not a problem the probe and/or filter heating system need not be used.) Allow time for the temperature to stabilize. If a Viton A O-ring or other leak free connection is used in assembling the probe nozzle to the probe liner, leak check the train at the sampling site by plugging the nozzle and pulling a 380 mm Hg (15 in. Hg) vacuum. (Note: A lower vacuum may be used provided that it is not exceeded during the test.) If an asbestos string is used, do not connect the probe to the train during the leak check. Instead, leak check the train as above by first plugging the inlet to the filter holder. Then connect the probe to the train and leak check at about 25 mm Hg (1 in. Hg) vacuum. A leakage rate in excess of 4% of the average sampling rate or 0.00057 m^3/min. (0.02 cfm), whichever is less, is unacceptable in either case.

The following leak check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with by-pass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the by-pass valve until 380 mm Hg (15 in. Hg) vacuum is reached. Do not reverse direction of by-pass valve. This will cause water to back up into the filter holder. If 380 mm Hg (15 in. Hg) is exceeded, either leak check at this higher vacuum or end the leak check as shown below and start over.

When the leak check is completed, first slowly remove the plug from the inlet to the probe or filter holder and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder and silica gel from being entrained backward into the third impinger.

Leak checks shall be conducted as described whenever the train is disengaged, e.g. for silica gel or filter changes during the test, prior to each test run, and at the completion of each test run. If leaks are found to be in excess of the acceptable rate, the test will be considered invalid. To reduce lost time due to leakage occurrences, it is recommended that leak checks be conducted between port changes.

4.1.5 Particulate train operation--During the sampling run, isokinetic sampling rate to within 10%, or as specified by the Administrator, of true isokinetic and the temperature around the filter of no greater than 120 ± 14° C (248 ± 25° F), or as specified by an applicable subpart of the standards, shall be maintained.

For each run, record the data required on the example data sheet shown in Figure 5-2. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, and when sampling is halted. Take other data point readings at least once at each sample point during each time increment and

PLANT ______________________

LOCATION ______________________

OPERATOR ______________________

DATE ______________________

RUN NO. ______________________

SAMPLE BOX NO. ______________________

METER BOX NO. ______________________

METER $\Delta H_@$ ______________________

C FACTOR ______________________

PITOT TUBE COEFFICIENT, C_p ______________________

SCHEMATIC OF STACK CROSS SECTION

AMBIENT TEMPERATURE ______________________

BAROMETRIC PRESSURE ______________________

ASSUMED MOISTURE, % ______________________

PROBE LENGTH, m (ft) ______________________

NOZZLE IDENTIFICATION NO. ______________________

AVERAGE CALIBRATED NOZZLE DIAMETER, cm (in.) ______________________

PROBE HEATER SETTING ______________________

LEAK RATE, m^3/min (cfm) ______________________

PROBE LINER MATERIAL ______________________

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	STATIC PRESSURE mm Hg (in Hg)	STACK TEMPERATURE (T_S) °C (°F)	VELOCITY HEAD (ΔP_S), mm(in.)H_2O	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER mm H_2O (in. H_2O)	GAS SAMPLE VOLUME m^3 (ft^3)	GAS SAMPLE TEMPERATURE AT DRY GAS METER		FILTER HOLDER TEMPERATURE, °C (°F)	TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F)
							INLET °C (°F)	OUTLET °C (°F)		
TOTAL							Avg.	Avg.		
AVERAGE							Avg.			

Figure 5-2. Particulate field data.

additional readings when significant changes (20% variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer.

Clean the portholes prior to the test run to minimize chance of sampling the deposited material. To begin sampling, remove the nozzle cap, verify that the filter and probe are up to temperature, and that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available for sampling trains using type S pitot tubes with 0.85 ± 0.02 coefficient and when sampling in air or a stack gas with equivalent density (molecular weight equal to 29 ± 4), which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. APTD-0576 details the procedure for using these nomographs. If C_p and M_d are outside the above stated ranges, do not use the nomograph unless appropriate steps are taken to compensate for the deviations.

When the stack is under significant negative stack pressure (height of impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to avoid water backing into the filter holder. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

Traverse the stack cross section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes to minimize chance of extracting deposited material.

During the test run, make periodic adjustments to keep the temperature around the filter holder at the proper temperature and add more ice and, if necessary, salt to maintain a temperature of less than 20° C (68° F) at the condenser/silica gel outlet to avoid excessive moisture losses. Also, periodically check the level and zero of the manometer.

If the pressure drop across the filter becomes too high making isokinetic sampling difficult to maintain, the filter may be replaced in the midst of a sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. After the new filter or filter assembly is installed conduct a leak check. The particulate weight shall include the summation of all filter assembly catches.

A single train shall be used for the entire sample run, except for filter and silica gel changes. However, if approved by the Administrator, two or more trains may be used for a single test run when there are two or more ducts or sampling ports. The results shall be the total of all sampling train catches.

At the end of the sample run, turn off the pump, remove the probe and nozzle from the stack, and record the final dry gas meter reading. Perform a leak check at a vacuum equal to or greater than the maximum reached during sampling. Calculate percent isokinetic (see calculation section) to determine whether another test run should be made. If there is difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

4.2 Sample recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period.

When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to prevent losing or gaining particulate matter. Do not cap off the probe tip tightly while the sampling train is cooling down as this would create a vacuum in the filter holder, thus drawing water from the impingers into the filter.

Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate, if present. Wipe off the silicone grease from the filter inlet where the probe was fastened and cap it. Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used between the first impinger or condenser and the filter holder, disconnect the line at the filter holder and let any condensed water or liquid drain into the impingers or condenser. After wiping off the silicone grease, cap off the filter holder outlet and impinger inlet. Either ground glass stoppers or plastic caps or serum caps may be used to close these openings.

Transfer the probe and filter-impinger assembly to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Save a portion of the acetone used for cleanup as a blank. Place about 200 ml of this acetone in a glass sample container labeled "acetone blank."

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

Container No. 1. Carefully remove the filter from the filter holder and place in its identified petri dish container. Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the particulate cake is inside the fold. Quantitatively remove any particulate matter and/or filter which adheres to the filter holder by carefully using a dry nylon bristle brush and/or a sharp-edged blade and place into this container. Seal the container.

Container No. 2. Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with acetone and placing the wash into a glass container in the following manner.

Distilled water may be used when approved by the Administrator or shall be used when specified by the Administrator. In these cases, save a water blank and follow Administrator's directions on analysis.

Carefully remove the probe nozzle and clean the inside surface by rinsing with acetone from a wash bottle and brushing with a nylon bristle brush. Brush until acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone.

Brush and rinse with acetone the inside parts of the Swagelok fitting in a similar way until no visible particles remain.

Rinse the probe liner with acetone by tilting the probe and squirting acetone into its upper end, while rotating the probe so that all inside surfaces will be rinsed with acetone. Let the acetone drain from the lower end into the sample container. A funnel may be used to aid in transferring liquid washes to the container. Follow the acetone rinse with a probe brush. Hold the probe in an inclined position, squirt acetone into the upper end as the probe brush is being pushed with a twisting action through the probe, hold a sample container underneath the lower end of the probe, and catch any acetone and particulate matter which is brushed from the probe. Run the brush through the probe three times or more until no visible particulate matter is carried out with the acetone or remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with acetone and quantitatively collect these washings in the sample container. After the brushing make a final acetone rinse of the probe as described above.

It is recommended that two people be used to clean the probe to minimize losing the sample. Between sampling runs, keep brushes clean and protected from contamination.

After ensuring that all joints are wiped clean of silicone grease, clean the inside of the front half of the filter holder by rubbing the surfaces with a nylon bristle brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. After all acetone washings and particulate matter are collected in the sample container, tighten the lid on the sample container so that acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label container to clearly identify its contents.

Container No. 3. Note color of indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to the original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the walls and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure under analysis.

Impinger water. Treat the impingers or condenser as follows: Make a notation of any color or film in the liquid catch. Measure the liquid which is in the first three impingers to within ± 1 ml by using a graduated cylinder or, if available, to within ± 0.5 g by using a balance. Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas.

If analysis of the impinger catch is not required, discard the liquid after measuring and recording the volume or weight. If analysis of the impinger catch is required, leave the impingers intact to transfer the liquid, cap off the inlet, and pour the liquid through the outlet into the graduated cylinder or into a sample container after its weight has been determined.

If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically.

4.3 Analysis. Record the data required on the example sheet shown in Figure 5-3. Handle each sample container as follows:

Container No. 1. Leave in shipping container or transfer the filter and any loose particulate from the sample container to a tared glass weighing dish and desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight and report the results to the nearest 0.1 mg. For purposes of this section 4.3, the term "constant weight" means a difference of no more than 0.5 mg or 1% of total weight less tare weight, whichever is greater, between two consecutive weighings, with no less than 6 hours of desiccation time between weighings and no more than 2 minutes exposure to the laboratory atmosphere (must be less than 50% relative humidity) during weighing.

Plant ____________________________

Date ____________________________

Run No. ____________________________

Relative Humidity ____________________________

Amount liquid lost during transport ____________________________

Acetone blank volume, ml ____________________________

Acetone wash volume, ml ____________________________

Acetone blank concentration, mg/mg (equation 5-4) ____________________________

Acetone wash blank, mg (equation 5-5) ____________________________

CONTAINER NUMBER	WEIGHT OF PARTICULATE COLLECTED, mg		
	FINAL WEIGHT	TARE WEIGHT	WEIGHT GAIN
1			
2			
TOTAL			

Less acetone blank	
Weight of particulate matter	

	VOLUME OF LIQUID WATER COLLECTED	
	IMPINGER VOLUME, ml	SILICA GEL WEIGHT, g
FINAL		
INITIAL		
LIQUID COLLECTED		
TOTAL VOLUME COLLECTED		g* ml

CONVERT WEIGHT OF WATER TO VOLUME BY DIVIDING TOTAL WEIGHT INCREASE BY DENSITY OF WATER (1g/ml).

$$\frac{\text{INCREASE, g}}{1\text{ g/ml}} = \text{VOLUME WATER, ml}$$

Figure 5-3. Analytical data.

Container No. 2. Note level of liquid in container and confirm on analysis sheet whether or not leakage occurred during transport. Measure the liquid in this container either volumetrically to ± 1 ml or gravimetrically to ± 0.5 g. Transfer the contents to a tared 250 ml beaker, and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

Container No. 3. Weigh the spent silica gel to the nearest 0.5 g using a balance. This step may be conducted in the field.

"Acetone Blank" Container. Measure acetone in this container either volumetrically or gravimetrically. Transfer the acetone to a tared 250 ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

5. Calibration

Maintain a laboratory log of all calibrations.

5.1 Probe nozzle. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Make 3 separate measurements using different diameters each time and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.).

When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use.

Each nozzle shall be permanently and uniquely identified.

5.2 Pitot tube. The pitot tube shall be calibrated according to the procedure outlined in Method 2.

5.3 Dry gas meter and orifice meter. Both meters shall be calibrated according to the procedure outlined in APTD-0576. When diaphragm pumps with by-pass valves are used, check for proper metering system design by calibrating the dry gas meter at an additional flow rate of 0.0057 m^3/min. (0.2 cfm) with the by-pass valve fully opened and then with it fully closed. If there is more than ± 2% difference in flow rates when compared to the fully closed position of the by-pass valve, the system is not designed properly and must be corrected.

5.4 Probe heater calibration. The probe heating system shall be calibrated according to the procedure contained in APTD-0576. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

5.5 Temperature gauges. Calibrate dial and liquid filled bulb thermometers against mercury-in-glass thermometers. New thermocouples need not be calibrated. Calibrate used thermocouples against new ones. For other devices, check with the Administrator.

6. Calculations

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

6.1 Nomenclature

A_n = Cross sectional area of nozzle, m^2 (ft^2)

B_{ws} = Water vapor in the gas stream, proportion by volume

C_a = Acetone blank residue concentration, mg/mg

c_s = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g/dscm (g/dscf)

I = Percent of isokinetic sampling

m_n = Total amount of particulate matter collected, mg.

M_w = Molecular weight of water, 18 g/g-mole (18 lb/lb-mole)

m_a = Mass of residue of acetone after evaporation, mg

P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg)

P_s = Absolute stack gas pressure, mm Hg (in. Hg)

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg)

R = Ideal gas constant, 0.06236 mm Hg-m^3/^{o}K-g-mole (21.83 in. Hg-ft^3/^{o}R-lb-mole)

T_m = Absolute average dry gas meter temperature (see Figure 5-2), ^{o}K (^{o}R)

T_s = Absolute average stack gas temperature (see Figure 5-2), ^{o}K (^{o}R).

T_{std} = Standard absolute temperature, 293^o K (528^o R)

V_a = Volume of acetone blank, ml

V_{aw} = Volume of acetone used in wash, ml

V_{lc} = Total volume of liquid collected in impingers and silica gel (see Figure 5-3, ml.

V_m = Volume of gas sample as measured by dry gas meter, dcm (dcf)

$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter corrected to standard conditions, dscm (dscf).

$V_{w(std)}$ = Volume of water vapor in the gas sample corrected to standard conditions, scm (scf).

v_s = Stack gas velocity, calculated by Method 2, Equation 2-7 using data obtained from Method 5, m/sec (ft/sec)

W_a = Weight of residue in acetone wash, mg

ΔH = Average pressure differential across the orifice (see Figure 5-2), meter, mm H_2O (in. H_2O)

ρ_a = Density of acetone, mg/ml (see label on bottle)

ρ_w = Density of water, 1 g/ml (0.00220 lb/ml)

θ = Total sampling time, min.

13.6 = Specific gravity of mercury

60 = sec/min

100 = Conversion to percent

6.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 5-2).

6.3 Dry gas volume. Correct the sample volume measured by the dry gas meter to standard conditions (20° C, 760 mm Hg or 68° F, 29.92 in. Hg) by using Equation 5-1.

$$V_{m(std)} = V_m \left(\frac{T_{std}}{T_m}\right) \left[\frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{std}}\right] = K\, V_m \left[\frac{P_{bar} + \Delta H/13.6}{T_m}\right]$$

Equation 5-1

where:

K = 0.3855 $^{\circ}$K/mm Hg for metric units

= 17.65 $^{\circ}$R/in. Hg for English units

6.4 Volume of water vapor.

$$V_{w(std)} = V_{lc}\left(\frac{\rho_w}{M_w}\right)\left(\frac{RT_{std}}{P_{std}}\right) = K\,V_{lc} \qquad \text{Equation 5-2}$$

where

$K = 0.00134\ m^3/ml$ for metric units

$= 0.0472\ ft^3/ml$ for English units

6.5 Moisture content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}} \qquad \text{Equation 5-3}$$

6.6 Acetone blank concentration.

$$C_a = \frac{m_a}{V_a\ \rho_a} \qquad \text{Equation 5-4}$$

6.7 Acetone wash blank.

$$W_a = C_a\ V_{aw}\ \rho_a \qquad \text{Equation 5-5}$$

6.8 Total particulate weight. Determine the total particulate catch from the sum of the weights obtained from containers 1 and 2 less the acetone blank (see Figure 5-3).

6.9 Particulate concentration.

$$c_s = (0.001\ g/mg)\ (m_n/V_{m(std)}) \qquad \text{Equation 5-6}$$

6.10 Conversion factors:

From	To	Multiply by
scf	m^3	0.0283
g/ft^3	gr/ft^3	15.4
g/ft^3	lb/ft^3	2.205×10^{-3}
g/ft^3	g/m^3	35.34

6.11 Isokinetic variation.

6.11.1 Calculations from raw data.

$$I = \frac{100\ T_s\ [K\ V_{1c} + (V_m/T_m)\ (P_{bar} + \Delta H/13.6)]}{60\ \theta\ v_s\ P_s\ A_n}$$

Equation 5-7

where:

$K = 0.00346$ mm Hg-m^3/ml-^oK for metric units

$= 0.00267$ in. Hg-ft^3/ml-^oR for English units

6.11.2 Calculations from intermediate values.

$$I = \frac{T_s\ V_{m(std)}\ P_{std}\ 100}{T_{std}\ v_s\ \theta\ A_n\ P_s\ 60\ (1-B_{ws})}$$

$$= K\ \frac{T_s\ V_{m(std)}}{P_s\ v_s\ A_n\ \theta\ (1-B_{ws})}$$

Equation 5-8

where:

$K = 4.323$ for metric units

$= 0.0944$ for English units

6.12 Acceptable results. If $90\% \leq I \leq 110\%$, the results are acceptable. If the results are low in comparison to the standards and I is beyond the acceptable range, the Administrator may option to accept the results. Use reference 7.4 to make judgments. Otherwise, reject the results and repeat the test.

7. References

7.1 Addendum to Specifications for Incinerator Testing at Federal Facilities, PHS, NCAPC, Dec. 6, 1967.

7.2 Martin, Robert M., Construction Details of Isokinetic Source Sampling Equipment, Environmental Protection Agency, APTD-0581.

7.3 Rom, Jerome J., Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment, Environmental Protection Agency, APTD-0576.

7.4 Smith, W. S., R. T. Shigehara, and W. F. Todd, A Method of Interpreting Stack Sampling Data, Paper presented at the 63d Annual Meeting of the Air Pollution Control Association, St. Louis, Mo., June 14-19, 1970.

7.5 Smith, W. S., et al., Stack Gas Sampling Improved and Simplified with New Equipment, APCA paper No. 67-119, 1967.

7.6 Specifications for Incinerator Testing at Federal Facilities, PHS, NCAPC, 1967.

APPENDIX B ILLUSTRATED USE OF NOMOGRAPHS

The material in this appendix is, in the most part, reproduced from APTD-0576 (ref. 9).

NOMOGRAPHS

The correction factor nomograph (fig. A1) and the operating nomograph (fig. A2) have been designed for use with the sampling train as aids for rapid isokinetic sampling rate adjustments and for selection of proper nozzle size. To determine the correction factor, C, on the nomograph, the following information is first required:

(1) Percent moisture, $\%H_2O$. This may be determined from a previous test or presurvey, or before the sample run.

(2) Orifice calibration factor, ΔH@. This is determined from the laboratory calibration (see section on Calibration).

(3) Meter temperature, T_m. Temperature at the meter rises above ambient temperature because of the pump and can easily be estimated with experience. An estimate within 10°F (approximately ± 1 percent error) is all that is necessary (an initial estimate of about 25°F above ambient temperature has been used). This approximation above ambient temperature is not required if the pump is located outside the meter box console.

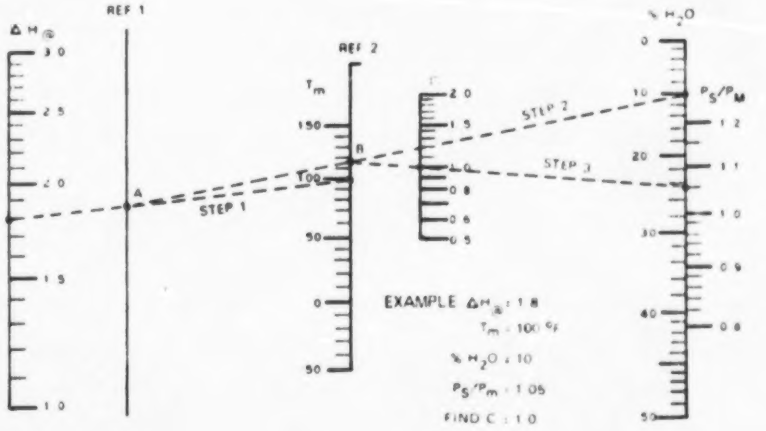

ΔH@
3.0
2.5
2.0
1.5
1.0
REF 1
A
STEP 1
REF 2
Tm
150
100
50
0
50
B
C
2.0
1.5
1.0
0.8
0.6
0.5
STEP 2
STEP 3
% H2O
0
10
20
30
40
50
PS/PM
1.2
1.1
1.0
0.9
0.8
EXAMPLE ΔH@ : 1.8
Tm : 100 °F
% H2O : 10
PS/Pm : 1.05
FIND C : 1.0

Figure A1. Nomograph for correction factor, C.

Preceding page blank

(4) Stack pressure, P_s. This is measured before the sample run; or if the sampling site is near the exit of the stack, atmospheric pressure is used.

(5) Meter pressure, P_m. Same as atmospheric pressure.

To obtain correction factor, C (fig. A1):

(1) Draw line from ΔH@ to T_m to obtain point "A" on reference line 1 (ref. 1).

(2) Draw line from point "A" to $\%H_2O$ to obtain point "B" on reference line 2 (ref. 2).

(3) Draw line from point "B" to the calculated value P_s/P_m to obtain correction factor, C.

To select the nozzle size and to set the K-factor on the operating nomograph, the following information is first required:

(1) C factor. This is obtained from the correction-factor nomograph (fig. A1).

Note: If the coefficient, C_p, of the type-S pitot tube is not equal to .85 ± .02, the following is required: (a) Multiply C times $\left(\frac{C_p}{.85}\right)$ = C' for the correct C factor in obtaining the K-factor, or (b) if C' is less than 0.5, then use C and multiply each ΔP reading by $\left(\frac{C_p}{.85}\right)^2$ for each adjustment.

(2) Stack temperature, T_s. This is determined in °F by a rough temperature traverse to within ± 25°F before the sample run.

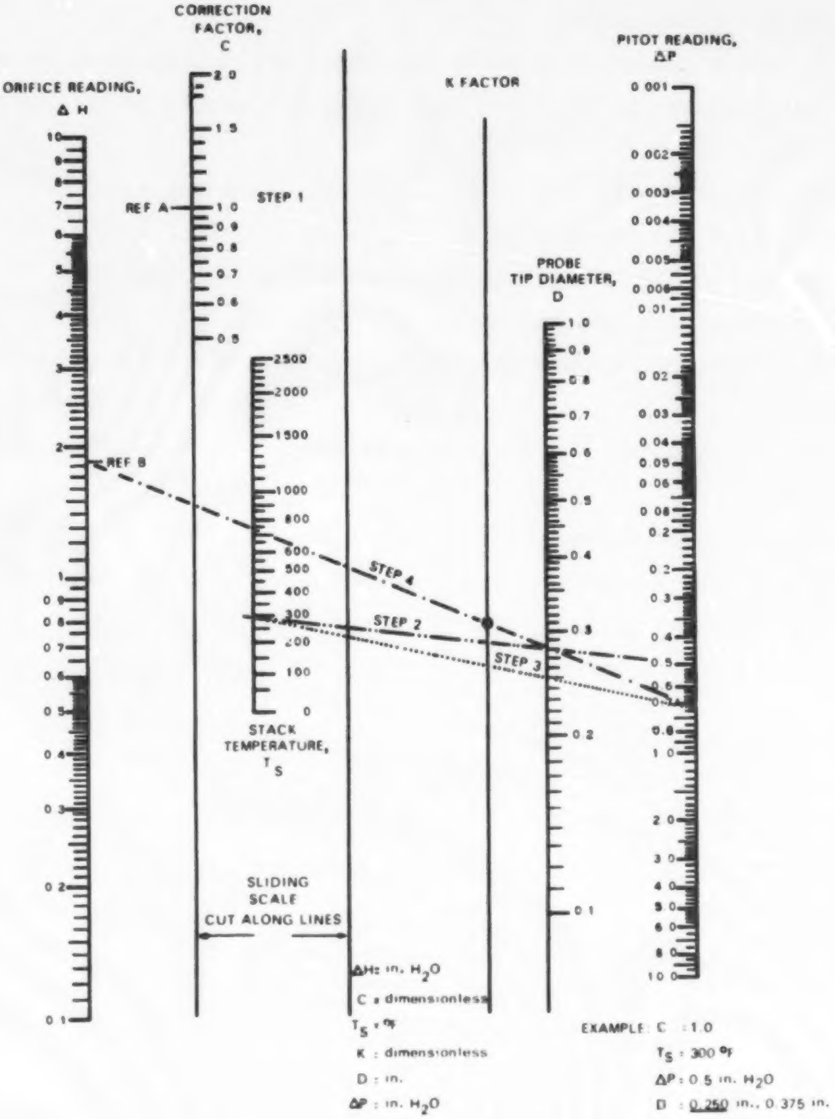
(3) Average velocity pressure, ΔP. This is determined by a rough preliminary pitot traverse, using the average of minimum and maximum ΔP's in inches of water.

(4) Exact available nozzle sizes, D. This is obtained from the calibration of available nozzles.

To select the nozzle size and to set the K-factor pivot point, use the following procedure (fig. A2):

(1) Set correction factor, C, on sliding scale to the reference mark, "A."

(2) Align T_s with average ΔP, note probe tip diameter on D-scale, and select exact nozzle size closest to it.


CORRECTION FACTOR, C
ORIFICE READING, Δ H
K FACTOR
PITOT READING, ΔP
PROBE TIP DIAMETER, D
STACK TEMPERATURE, T_S
REF A
REF B
STEP 1
STEP 2
STEP 3
STEP 4
SLIDING SCALE
CUT ALONG LINES
10
9
8
7
6
5
4
3
2
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
2.0
1.5
1.0
0.9
0.8
0.7
0.6
0.5
2500
2000
1500
1000
800
600
500
400
300
200
100
0
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.001
0.002
0.003
0.004
0.005
0.006
0.01
0.02
0.03
0.04
0.05
0.06
0.08
0.1
0.2
0.3
0.4
0.5
0.6
0.8
1.0
2.0
3.0
4.0
5.0
6.0
8.0
10.0

ΔH: in. H_2O
C : dimensionless
T_S : °F
K : dimensionless
D : in.
ΔP : in. H_2O

EXAMPLE: C : 1.0
T_S : 300 °F
ΔP : 0.5 in. H_2O
D : 0.250 in., 0.375 in.

Figure A2. Use of the nomograph in selecting nozzle size and setting K factor.

(3) Align T_s with exact nozzle size selected and obtain a value on the ΔP scale.

(4) Align the ΔP value with reference mark, "B", on ΔH scale, and set the K-factor pivot point.

To obtain the orifice meter settings, ΔH, for isokinetic conditions after the K-factor pivot point has been set, use the following procedure (fig. A3):

(1) Position the pitobe nozzle at the sampling point.

(2) Read the pitot tube ΔP.

(3) Align the ΔP through the K-factor pivot point

(4) Obtain ΔH and adjust metering valves.

The nomograph assumes the following, once the K-factor pivot point is set:

(1) T_s does not change more than 25° for $T_s < 1000°F$ or 50° for $T_s > 1000°F$.

(2) D is not changed during the test.

(3) T_m was estimated correctly and does not vary more than 10°.

(4) Percent H_2O remains constant, within $\pm$ 1.0 percent.

(5) P_s and P_m remain constant, within $\pm$ 1.0 percent.

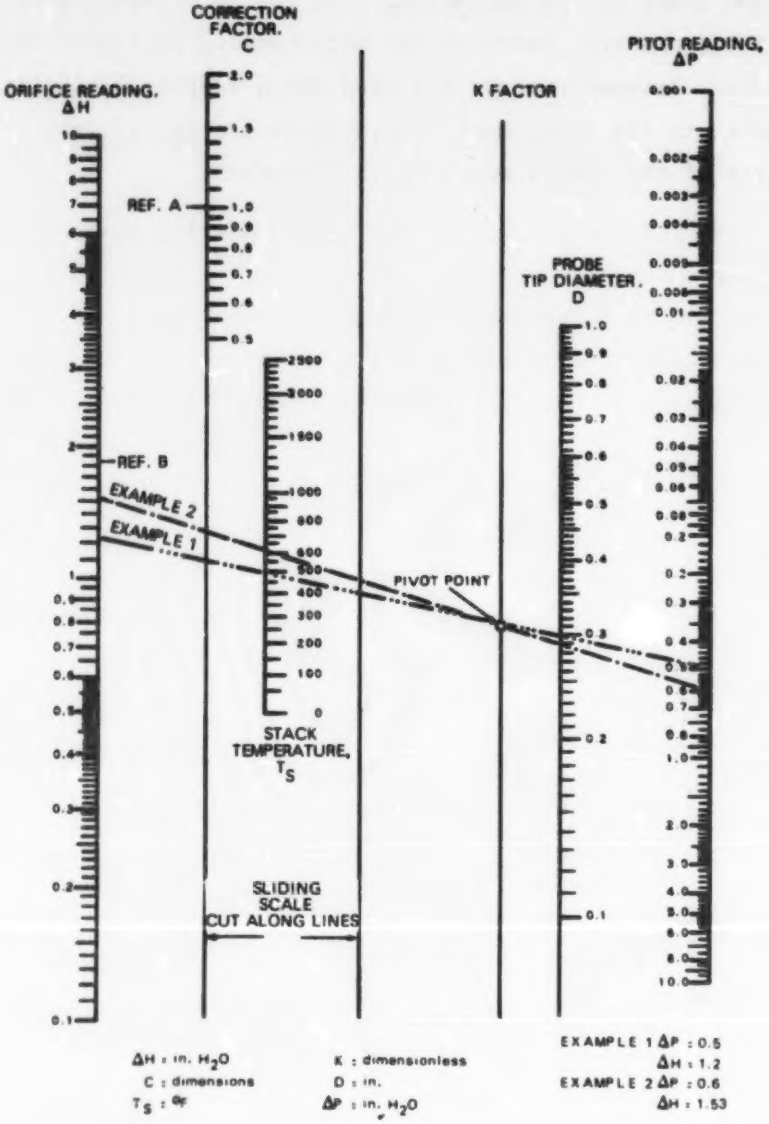

CORRECTION FACTOR, C
ORIFICE READING, ΔH
K FACTOR
PITOT READING, ΔP
PROBE TIP DIAMETER, D
STACK TEMPERATURE, T_S
REF. A
REF. B
EXAMPLE 2
EXAMPLE 1
PIVOT POINT
SLIDING SCALE CUT ALONG LINES
10
9
8
7
6
5
4
3
2
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
2.0
1.5
1.0
0.9
0.8
0.7
0.6
0.5
2500
2000
1500
1000
800
600
500
400
300
200
100
0
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.001
0.002
0.003
0.004
0.005
0.006
0.01
0.02
0.03
0.04
0.05
0.06
0.08
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
1.0
2.0
3.0
4.0
5.0
6.0
8.0
10.0
ΔH : in. H_2O
C : dimensions
T_S : °F
K : dimensionless
D : in.
ΔP : in. H_2O
EXAMPLE 1 ΔP : 0.5
ΔH : 1.2
EXAMPLE 2 ΔP : 0.6
ΔH : 1.53

Figure A3. Nomograph operation to obtain desired orifice meter settings.

APPENDIX C ILLUSTRATED AUDIT PROCEDURES

A flow chart of the operations involved in an auditing program from first setting desired limits on the data quality to filing the results is given below. Assumed numbers are used and a sample calculation of an audit is performed in the flow chart. Each operation is referred to the section in the text of the report where it is discussed.

MANAGER

1. LIMITS FOR DATA QUALITY CAN BE SET BY WHAT IS DESIRED OR FROM THE NATURAL VARIABILITY OF THE METHOD WHEN USED BY TRAINED AND COMPETENT PERSONNEL. FOR THIS EXAMPLE, IT IS ASSUMED THAT CV{$\overline{\text{PMR}}$} = 9.3 PERCENT (subsec.4.1), AND USING ± 3 CV{$\overline{\text{PMR}}$}, THE LIMITS ARE L = -28 PERCENT AND U = 28 PERCENT.

2. FROM PRIOR KNOWLEDGE OF DATA QUALITY, ESTIMATE THE PERCENTAGE OF FIELD MEASUREMENTS FALLING OUTSIDE THE ABOVE LIMITS. IF NO INFORMATION IS AVAILABLE, MAKE AN EDUCATED GUESS. IT IS ASSUMED IN THIS EXAMPLE THAT 50 PERCENT OF THE FIELD DATA ARE OUTSIDE THE LIMITS L AND U (subsec. 4.4.4).

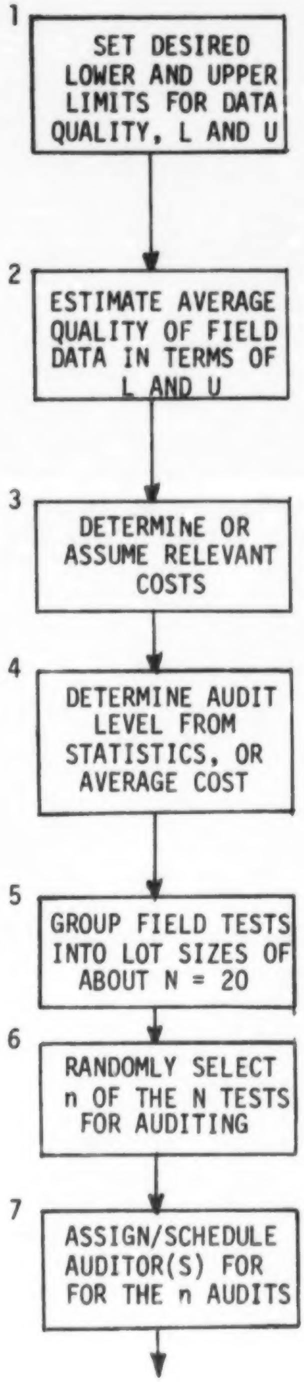
3. DETERMINE: (1) COST OF CONDUCTING AN AUDIT, (2) COST OF FALSELY INFERRING THAT GOOD DATA ARE BAD, (3) COST OF FALSELY INFERRING THAT BAD DATA ARE GOOD, AND (4) COST SAVINGS FOR CORRECTLY IDENTIFYING BAD DATA (subsec. 4.4.4).

4. DETERMINE THE AUDIT LEVEL EITHER BY (1) MINIMIZING AVERAGE COST USING EQUATION (41) OF SUBSECTION 4.4.4, OR (2) ASSURING A DESIRED LEVEL OF CONFIDENCE IN THE REPORTED DATA THROUGH STATISTICS. FOR THIS EXAMPLE, THE AUDIT LEVEL IS TAKEN AS n = 5 (fig. 15).

5. BY TEAMS, TYPES OF SOURCES, OR GEOGRAPHY, GROUP FIELD TESTS INTO LOTS (GROUPS) OF ABOUT 20 THAT WILL BE PERFORMED IN A PERIOD OF ONE CALENDAR QUARTER.

6. SELECT n OF THE N TESTS FOR AUDITING. COMPLETE RANDOMIZATION MAY NOT BE POSSIBLE DUE TO AUDITOR'S SCHEDULE. THE PRIMARY POINT IS THAT THE FIELD TEAM SHOULD NOT KNOW IN ADVANCE THAT THEIR TEST IS TO BE AUDITED.

7. ASSIGN OR SCHEDULE AN AUDITOR FOR EACH FIELD TEST.


1
SET DESIRED LOWER AND UPPER LIMITS FOR DATA QUALITY, L AND U
2
ESTIMATE AVERAGE QUALITY OF FIELD DATA IN TERMS OF L AND U
3
DETERMINE OR ASSUME RELEVANT COSTS
4
DETERMINE AUDIT LEVEL FROM STATISTICS, OR AVERAGE COST
5
GROUP FIELD TESTS INTO LOT SIZES OF ABOUT N = 20
6
RANDOMLY SELECT n OF THE N TESTS FOR AUDITING
7
ASSIGN/SCHEDULE AUDITOR(S) FOR FOR THE n AUDITS

AUDITOR

8. THE AUDITOR OBTAINS APPROPRIATE CALIBRATED EQUIPMENT AND SUPPLIES FOR THE AUDIT (subsec. 4.3).

9. OBSERVE THE FIELD TEAM'S PERFORMANCE OF THE FIELD TEST. FILL IN THE AUDITOR'S CHECKLIST (table 8) AND NOTE ANY UNUSUAL CONDITIONS THAT OCCURRED DURING THE TEST.

10. IN THE FIELD TEAM'S HOME LABORATORY, MAKE INDEPENDENT DETERMINATIONS OF C_p, D_n, γ, AND $\Delta H@$ (subsec. 4.3) ACCORDING TO THE CALIBRATION PROCEDURES GIVEN IN SUBSECTION 2.2.

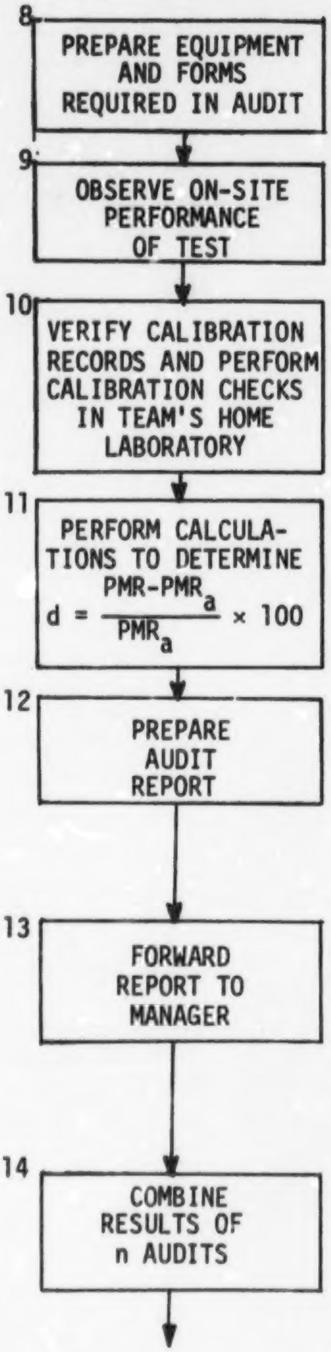
11. STARTING WITH THE RAW DATA FROM THE FIELD AND USING AUDIT VALUES, PERFORM ALL THE CALCULATIONS NECESSARY TO ARRIVE AT A VALUE FOR d (subsec. 4.3, fig. 10).

12. THE AUDITOR'S REPORT SHOULD INCLUDE (1) DATA SHEET FILLED OUT BY THE FIELD TEAM (fig. 3), (2) AUDITOR'S CHECKLIST WITH COMMENTS (table 8), (3) AUDIT DATA SHEET WITH CALCULATIONS (fig. 11), and (4) A SUMMARY OF THE TEAM'S PERFORMANCE WITH A NUMERICAL RATING (subsec. 4.3).

13. THE AUDITOR'S REPORT IS FORWARDED TO THE MANAGER.

MANAGER

14. COLLECT THE AUDITOR'S REPORTS FROM THE n AUDITS OF THE LOT OF N STACKS. IN THIS CASE $n = 5$ AND ASSUMED VALUES FOR THE AUDITS ARE $d_1 = 12.0$, $d_2 = -6.0$, $d_3 = 3.0$, $d = 15.0$ AND $d_5 = 9.0$ (table 8).


8
PREPARE EQUIPMENT AND FORMS REQUIRED IN AUDIT
9
OBSERVE ON-SITE PERFORMANCE OF TEST
10
VERIFY CALIBRATION RECORDS AND PERFORM CALIBRATION CHECKS IN TEAM'S HOME LABORATORY
11
PERFORM CALCULATIONS TO DETERMINE
$d = \frac{PMR-PMR_a}{PMR_a} \times 100$
12
PREPARE AUDIT REPORT
13
FORWARD REPORT TO MANAGER
14
COMBINE RESULTS OF n AUDITS

15. CALCULATE $\bar{d}$ AND s_d ACCORDING TO THE SAMPLE IN TABLE 10. RESULTS OF THIS SAMPLE CALCULATION SHOW $\bar{d}$ = 6.6, AND s_d = 8.3 (table 8, subsec. 4.4.3).

16. USE A t-TEST TO CHECK $\bar{d}$ FOR SIGNIFICANCE, FOR THIS EXAMPLE $t = 6.6 \times \sqrt{5}/8.3 = 1.78$. THE TABULATED t-VALUE FOR 4 DEGREES OF FREEDOM AT THE 0.05 LEVEL IS 2.13; HENCE, $\bar{d}$ IS NOT SIGNIFICANTLY DIFFERENT FROM 0 AT THIS LEVEL. ALSO, s_d IS CHECKED AGAINST THE ASSUMED VALUE OF 9.3 PERCENT BY A CHI-SQUARE TEST.

$$\chi^2/f = s_d^2/\sigma^2\{\bar{d}\} = (8.3)^2/(9.3)^2 = 0.797$$

THE TABULATED VALUE OF $\chi^2/4$ AT THE 95 PERCENT LEVEL IS 0.711; HENCE, s_d IS SIGNIFICANTLY DIFFERENT FROM 9.3 PERCENT (In this case it is a significant improvement).

17. OBTAIN THE VALUE OF k FROM TABLE 9, FOR n = 5 AND p = 0.1. THIS VALUE IS 2.742, THEN $\bar{d} + k\ s_d = 6.6 + 2.742(8.3) = 29.4$ AND $\bar{d} = k\ s_d = 6.6 - 2.742(8.3) = -16.2$ (subsec. 4.4.3).

18. COMPARE THE ABOVE CALCULATIONS WITH LIMITS L AND U (subsec. 4.4.3). FOR THIS EXAMPLE

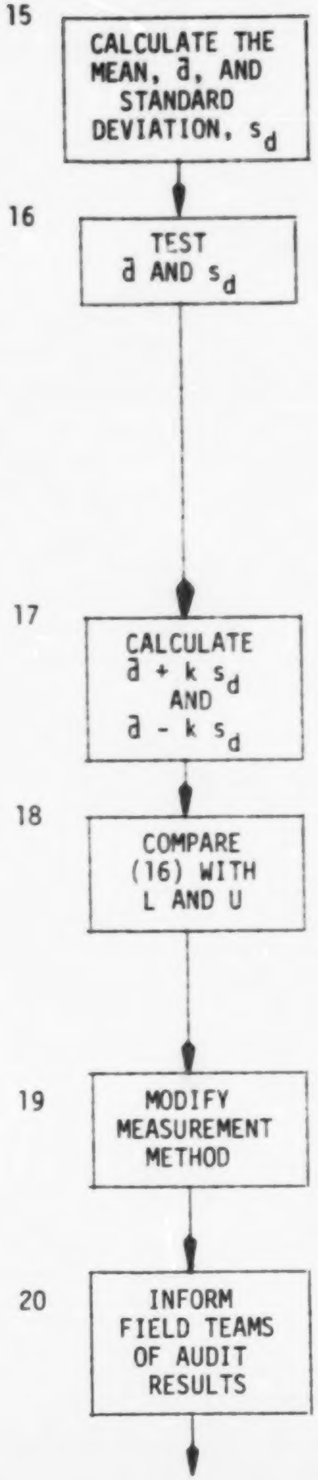
$$\bar{d} + k\ s_d = 29.4 > U = 28$$

$$\bar{d} - k\ s_d = -16.2 > L = -28.$$

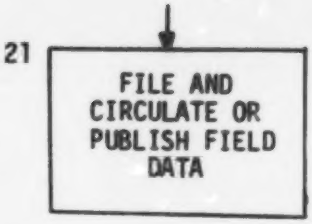
THE UPPER LIMIT U HAS BEEN EXCEEDED. BOTH $\bar{d}$ AND s_d WERE ACCEPTABLE INDIVIDUALLY BUT WHEN COMBINED THE UPPER LIMIT WAS EXCEEDED.

19. STUDY THE AUDIT AND FIELD DATA FOR SPECIFIC AREAS OF VARIABILITY, SELECT THE MOST COST EFFECTIVE ACTION OPTION(S) THAT WILL RESULTS IN GOOD QUALITY DATA (subsec. 4.2). NOTIFY THE FIELD TEAMS TO IMPLEMENT THE SELECTED ACTION OPTION(S).

20. A COPY OF THE AUDITOR"S REPORT SHOULD BE SENT TO THE RESPECTIVE FIELD TEAM. ALSO, THE DATA ASSESSMENT RESULTS, i.e., CALCULATED VALUES OF $\bar{d}$, s_d, AND COMPARISON WITH THE LIMITS L AND U SHOULD BE FORWARDED TO EACH TEAM INVOLVED IN THE N FIELD TESTS.


15
CALCULATE THE MEAN, $\bar{d}$, AND STANDARD DEVIATION, s_d
16
TEST $\bar{d}$ AND s_d
17
CALCULATE $\bar{d} + k\ s_d$ AND $\bar{d} - k\ s_d$
18
COMPARE (16) WITH L AND U
19
MODIFY MEASUREMENT METHOD
20
INFORM FIELD TEAMS OF AUDIT RESULTS

21. THE FIELD DATA WITH AUDIT RESULTS ATTACHED ARE FILED. THE AUDIT DATA SHOULD REMAIN WITH THE FIELD DATA FOR ANY FUTURE USES.


21
FILE AND CIRCULATE OR PUBLISH FIELD DATA

APPENDIX D GLOSSARY OF SYMBOLS

This is glossary of symbols as used in this document. Symbols used and defined in the reference method (appendix A) are not repeated here.

SYMBOL	DEFINITION
N	Lot size, i.e., the number of field tests to be treated as a group.
n	Sample size for the quality audit (section IV).
$CV\{X\}$	Assumed or known coefficient of variation (100 σ_X/μ_X)
$\hat{CV}\{X\}$	Computed coefficient of variation (100 $s_X/\bar{X}$) from a finite sample of measurements.
$\sigma\{X\}$	Assumed standard deviation of the parameter X (population standard deviation).
$s\{X\}$	Computed standard deviation of a finite sample of measurements (sample standard deviation)
$\hat{\tau}\{X\}$	Computed bias of the parameter X for a finite sample (sample basis).
R	Range, i.e., the difference in the largest and smallest values in r replicate analyses.
$\varepsilon\{M_n\}$	Random error associated with the measurement of particulate mass, M_n.
d_j	The percent difference in the audit value and the value of $\overline{PMR}$ arrived at by the field crew for the j^{th} audit.
$\bar{d}$	Mean difference between PMR_j and PMR_{aj} for n audits expressed as a percent.
s_d	Computed standard deviation of differences between PMR_j and PMR_{aj} expressed as a percent.
p	Percent of measurement outside specified limits L and U.
k	Constant used in sampling by variables (section IV).
$P\{Y\}$	Probability of event Y occurring
$t_{(n - 1)}$	Statistic used to determine if the sample bias, $\bar{d}$, is significantly different from zero (t-test).
$\chi^2/(n - 1)$	Statistic used to determine if the sample variance, s^2, is significantly different from the assumed variance, σ^2, of the parent distribution (chi-square test)

APPENDIX D GLOSSARY OF SYMBOLS (CONTINUED)

SYMBOL	DEFINITION
L	Lower quality limit used in sampling by variables.
U	Upper quality limit used in sampling by variables.
CL	Center line of a quality control chart.
LCL	Lower control limit of a quality control chart.
UCL	Upper control limit of a quality control chart.
PMR	Particulate mass emission rate reported by the field team for a sample run; it could be either PMR_c or $1/2(PMR_c + PMR_a)$.
PMR_c	Particulate mass emission rate calculated by the sample concentration method, i.e., $PMR_c = C_s \times Q_s$, g/hr.
PMR_a	Particulate mass emission rate calculated by the ratio of area method, i.e., $PMR_a = \frac{M_n}{\theta} \frac{A_s}{A_n}$, g/hr.
$\overline{PMR}$	Average particulate mass emission rate for a field test, i.e., the average of three sample runs, g/hr.

APPENDIX E GLOSSARY OF TERMS

The following glossary lists and defines the statistical terms as used in this document.

Accuracy	A measure of the error of a process expressed as a comparison between the average of the measured values and the true or accepted value. It is a function of precision and bias.
Bias	The systematic or nonrandom component of measurement error.
Lot	A specified number of objects to be treated as a group, e.g., the number of field tests to be conducted by an organization during a specified period of time.
Measurement method	A set of procedures for making a measurement.
Measurement process	The process of making a measurement including method, personnel, equipment, and environmental conditions.
Population	A large number of like objects (i.e., measurements, checks, etc.) from which the true mean and standard deviation can be deduced with a high degree of accuracy.
Precision	The degree of variation among successive, independent measurements (e.g., on a homogeneous material) under controlled conditions, and usually expressed as a standard deviation or, as is done here, as a coefficient of variation.
Quality audit	A management tool for independently assessing data quality.
Quality control check	Checks made by the field crew on certain items of equipment and procedures to assure data of good quality.
Sample	Objects drawn, usually at random, from the lot for checking or auditing purposes.

APPENDIX F CONVERSION FACTORS

Conversion factors for converting the U.S. customary units to the International System of Units (SI)[*] are given below.

To Convert from	To	Multiply by
	Length	
foot	meter (m)	0.3048
inch	meter (m)	0.0254
	Pressure	
inch of mercury (in. of Hg) (32°F)	Newton/meter^2 (N/m^2)	3386.389
inch of mercury (in. of Hg) (60°F)	Newton/meter^2 (N/m^2)	3376.85
millimeter mercury (mmHg) (32°F)	Newton/meter^2 (N/m^2)	133.3224
inch of water (in. of H_2O) (29.2°F)	Newton/meter^2 (N/m^2)	249.082
inch of water (in. of H_2O) (60°F)	Newton/meter^2 (N/m^2)	248.84
	Force	
pound-force (lbf avoirdupois)	Newton (N)	4.448222
	Mass	
pound-mass (lbm avoirdupois)	kilogram (kg)	0.4535924
	Temperature	
degree Celsius	kelvin (K)	$t_K = t_C + 273.15$
degree fahrenheit	kelvin (K)	$t_K = (t_F+459.67)/1.8$
degree rankine	kelvin (K)	$t_K = t_R/1.8$
degree fahrenheit	degree Celsius	$t_C = (t_F - 32)/1.8$
kelvin	degree Celsius	$t_C = t_K - 273.15$
	Velocity	
foot/second	meter/second (m/s)	0.3048
foot/minute	meter/second (m/s)	0.00508
	Volume	
cubic foot (ft^3)	meter^3 (m^3)	0.02832
	Volume/Time	
foot^3/minute	meter^3/second (m^3/s)	0.0004719
foot^3/second	meter^3/second (m^3/s)	0.02832

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[*] Metric Practice Guide (A Guide to the Use of SI, the International Systems of Units), American National Standard Z210.1-1971, American Society for Testing and Materials, ASTM Designation:E380-70, Philadelphia, Pa., 1971.